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*N/R = No Report.

PROJECT NUMBER: 2107
PROJECT TITLE: Filter Research & Development
PROJECT LEADER: C. J. Campbell
PERIOD COVERED: October, 1988

I. FILTER RESEARCH & DEVELOPMENT GROUP:

- A. **Objective:** Consolidate efforts on filter research and development into one group and actively pursue the development of improved filter systems.
- B. **Status:** Concentric filters which selectively filter either core smoke or peripheral smoke are being investigated to determine whether they have a subjective advantage over conventional filters. Filtrona is producing six models of concentric filters, three core flow and three peripheral flow, for evaluation on a Merit Ultra Lights type cigarette. These will be subjectively compared to a control MUL at the same tar delivery.

A computer model of the concentric filter has been developed and is being used in design cigarette. Two Marlboro Lights type cigarettes with peripheral flow concentric filters were constructed and evaluated analytically. The information obtained will be used to improve the computer model.

A Sun work station has been installed in our lab and will allow improved access to cigarette performance modeling.

Communications with FTR are being maintained on projects of mutual interest such as concentric filters and CA web.

II. FILTER SUPPORT FOR MAJOR PROGRAMS:

- A. **Objective:** Provide design assistance and potential new filter systems for major R&D programs.
- B. **Status:** Four filter samples have been attached to rods containing two different blends of ART filler and are being subjectively evaluated by the Flavor Panel. These consist of a core flow concentric filter, a peripheral flow concentric filter, a filter with potassium carbonate additive, and a filter with dipotassium phosphate additive. Additional ART blends, filters, and menthol samples will be evaluated.

New formulations of PVA Menthol which use Benzoflex plasticizer and result in a lower alcohol content have been mixed and are being tested for menthol retention. An attempt is also being made to increase the menthol content per unit volume to simplify application.

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III. CELLULOSE ACETATE WEB:

- A. Objective: Explore the potential advantages of CA web for use as a high efficiency filter material.
- B. Status: Celanese has now received the CA web material from Dexter and is in the process of making filter rods for our evaluation.
- C. Plans: Evaluate samples for efficiency and subjectives.

IV. IMPROVED MENTHOL STABILITY:

- A. Objective: Investigate methods of improving the stability of menthol delivery in smoke of aged cigarettes.
- B. Status: An aging study of menthol cigarettes made from Filtrona heat set filter rods is continuing into week five. Menthol content of the filter and delivery of menthol in smoke is being compared with a control cigarette to test the ability of the heat set filter to resist menthol absorption during aging.

Two models of Marlboro Lights Menthol cigarettes with PVA menthol and a control which has a standard filter and mentholated foil were made and evaluated. Analytical results showed that one of the PVA models was low on menthol delivery. Subjective evaluation by a Flavor Panel indicated a definite preference for the mentholated foil model. A second subjective evaluation by an OC or MC panel is planned.

V. TOW VENDOR R&D COORDINATION:

- A. Objective: Coordinate specific requests for R&D work by Eastman and Celanese.
- B. Status: Eastman 1.6 dpf filters and Filtrona UHF filters are on hand awaiting cigarette fabrication by Semiworks. These may allow the construction of ultra low delivery cigarettes with reduced ventilation. A more comprehensive test is being planned to include subjective evaluation.

An annual review of Celanese R&D efforts has been scheduled for December 7.

VI. FILTER ADDITIVES:

- A. Objective:** Investigate the effects of filter additives for analytical and subjective benefits.
- B. Status:** Six models of GCC Marlboro cigarettes made with filters containing various additives were made in the Semiworks and have been analytically tested. Filters made with citric acid added and those with lactic acid added (by Celanese) showed a good reduction (15%) in nicotine/tar ratio. These additives present a problem in application, however, requiring a separate application booth on the plugmaker. Another additive, diethyl citrate, also showed a reasonable reduction (12%) in nicotine/tar ratio, but it can be applied along with triacetin. Filter firmness and subjectives are now being evaluated.

Additional screening of FML filters with hand applied additives has shown that significant modification of subjectives is possible. Samples of these additives are on hand and machine made plugs with consistently applied additives have been requested from the FML group for subjective evaluation.

- C. Plans:** Produce FML filters with additives and evaluate.

PROJECT NUMBER: 2108
PROJECT TITLE: New Product Technology
PROJECT LEADER: W. T. Callahan
PERIOD COVERED: October, 1988

I. PROJECT SAUNA

- A. **Objective:** Develop an acceptable product with a modified plastic fluted filter.
- B. **Status:** Cigarette models were prepared with reduced smoke back-flow while maintaining the ISO tar level at approximately the same as for Saudi Barclay. This was achieved by reducing the amount of front band dilution and electrostatically perforating the cigarette paper. These models had front band dilution levels of 30% (laser), 12% (ESP), and 0% the paper porosities of these models were 50, 77, 132, 154, and 170 Coresta. One model had a low pressure drop filter.

The models with 0% front band dilution give no smoke back flow while those with 12% dilution had less than for Saudi Barclay. Subjective screening by Flavor Development rated the 12% dilution models first. The preferred model had 132 Coresta cigarette paper. The deliveries of the preferred models and Barclay were as follows:

	<u>BARCLAY</u>		<u>SAUNA</u>
FRONT BAND DILUTION, %	12	12	~32
PAPER POROSITY, Coresta	132	50	~25
ISO TAR, mg	11.7	11.6	11.7
PUFF COUNT	6.9	6.8	8.2

- C. **Plans:** The preferred models will be remade using cork-on-white tipping (to facilitate ESP front band dilution), analytically tested, and submitted to EEMA for evaluation.

II. HUMIDOR PACK

- A. **Objective:** Develop a moisture release device for use in a cigarette pack which maintains the pack OV at a desired level.
- B. **Status:** Celanese (manufacturer of Celgard) has recommended a contract packager (Paco Pharmaceutical Services) who has experience producing packets with Celgard. A confidentiality agreement was signed and discussions were held with Paco. Paco indicated that they would have similar problems with the current packet design as were observed at Klockner Packaging (edge curling and leaking), so a modified packet was suggested. This packet will allow foil-to-foil sealing and will have a Celgard "window" on one side to allow water vapor transmission. This should eliminate curling.

A meeting was held with Physical Research to initiate a study to determine if the humidor solution can be reformulated. Packet formation, packet insertion into the cigarette pack, and seal quality could be improved by eliminating or reducing the requirement of excess salt in the solution.

Aging trials of cellulose triacetate film with the improved foil laminate backing were begun, however, leaking seals occurred after one day, thereby halting the study. Reynolds Metals has been requested to provide an acceptable EVA laminate backing which will not fail at high temperatures.

Installation of the modified GDX-2 packer in the OC basement has been delayed due to PM Engineering priorities.

- C. **Plans:** Initial trials of the Celgard window packet are scheduled for early to mid-December. This timing allows for a die to be fabricated to produce the Celgard window and components to be in place for producing machine-made packets. PM Engineering and R&D will visit Paco next week to finalize packet design and to discuss their solution dispensing capabilities.

III. KAYMICH MENTHOL APPLICATOR

- A. **Objective:** Evaluate a Kaymich menthol applicator as an alternative mentholation process.
- B. **Status:** A study to analytically and subjectively compare several Kaymich menthol prototypes with a menthol-on-foil control has been initiated. Kaymich prototypes at four menthol levels were made and submitted for testing. During this run, a modified 8-hole nozzle applicator was used which distributed the menthol over a larger area of paper to minimize bleed-through. Excess menthol buildup at the paper guide was observed and menthol losses at the maker were approximately the same as in previous trials (below):

	MOF	KAYMICH			
TARGET PACK MENTHOL, mg	76	86	82	78	74
ACTUAL PACK MENTHOL, mg	75.3	77.0	76.0	71.0	70.5
MENTHOL LOSS, %	-0.9	-10.5	-7.3	-9.0	-4.7

- C. **Plans:** Complete the analytical and subjective testing and make a recommendation regarding further evaluation of the Kaymich system.

IV. EMBOSSING TECHNOLOGY

- A. **Objective:** Explore embossing technology for potential new product development.
- B. **Status:** The second generation laboratory embossing unit has been delivered and is currently being debugged.
- C. **Plans:** Continue providing support to Engineering as needed and pursuing other applications for new product development.

V. MENTHOL ON FOIL

- A. Objective: Support the introduction of the menthol on foil process..
- B. Status: Mentholated foils were prepared for Domestic and International Product Development and Cigarette Technology studies during the month of October.

The third mentholator control problem has not been corrected yet by Engineering due to priorities.

- C. Plans: Produce samples as requested. Qualify the third mentholator for use in the Semiworks when operational.

PROJECT NUMBER: 2305
PROJECT TITLE: Flavor Development-Brand Modification-Menthol/Distinctive
PROJECT LEADER: H. M. Maxwell
PERIOD COVERED: October, 1988

I. ALTERNATIVE FLAVORS:

- A. Objective: To identify and qualify alternative sources for flavoring materials.
- B. Status: POL testing of alternative Jonex is in progress. Cigarettes for a replicate test were remade and approved for testing.
- C. Plans:

POL Results For Alternative October, 1988
Jonex (Merit 85)

Replicate POL Results November, 1988

II. PROJECT MOOG:

- A. Objective: To develop the expertise to produce cigarettes that are subjectively equivalent to Salem, Newport and Koal.
- B. Status: A K-type POL test will be made when Semi-works scheduling permits. Two S-type models were made and will be evaluated if analytically acceptable. A POL test of N-type cigarettes versus Newport is in testing.
- C. Plans:

S-Type Flavor October, 1988
Development

N-type POL Results November, 1988

III. MENTHOL RELEASE COMPOUND:

- A. Objective: To develop a mentholated charcoal filtered cigarette utilizing a menthol release compound. Also, to apply menthol release technology to other areas.
- B. Status: The Foam Bound Rod group is continuing to investigate problems of MGC application. Poly menthol isopropenyl carbonate was extruded on the "DL" blend at levels up to 10% level. Evaluation is in progress. Samples of the anethole/menthol release compound were requested for further product development studies. These samples will not be available until Phase II clearance is obtained.
- C. Plans:

Cigarette Remake (MGC)

To be scheduled

IV. ALTERNATE HUMECTANT PROGRAM:

A. Objective: To develop the capability to produce cigarettes that are propylene glycol and glycerin free, while maintaining product subjective integrity.

B. Status: Efforts of this program were shifted to development of a PG/G-free acceptable cigarette. Two POL tests will be run to confirm subjective acceptability.

C. Plans:

PG/G-free POL's

To Be Scheduled

V. PROJECT GRAIN:

A. Objective: To significantly reduce alcohol levels in PM flavor systems, while maintaining product subjective integrity.

B. Status: POL testing is on hold. Aftercuts prepared at Twentieth Street to investigate low anethole levels have not resolved that problem. Further investigations will be conducted.

C. Plans:Cigarette Production
for Internal Testing

To Be Scheduled

Resolution of low
Anethole

November, 1988

VI. CASING STUDY:

A. Objective: To determine the effect of prolonged heating on burley spray and its ingredients, block chocolate and block licorice.

B. Status: Analyses of the condensates from the one-liter 24-hour trials indicate that insufficient quantities were collected. Further trials with twelve liter quantities will be run.

C. Plans:

Complete trial runs to further define scope and duration of study.

PROJECT NUMBER: 2306
PROJECT TITLE: Marlboro Standardization/International Support
PROJECT LEADER: Janet L. Spruill
PERIOD COVERED: October, 1988

I. MARLBORO STANDARDIZATION

- A. Objective: Analytical and subjective evaluations of production Marlboro KS/LS.
- B. Status: Factory pick-up from 7/19 complete. Standard Run VI for all factory locations is week of November 1.
- C. Plans: On-site participation for Standard Run VI.

II. DOMESTIC CIGARETTE DEVELOPMENT PANEL

- A. Objective: To provide subjective direction for programs within R&D and manufacturing locations.
- B. Status: ART panels run during reporting period.
- C. Plans: Provide assistance as needed.

III. PROJECT NATURAL

- A. Objective: To develop 85mm and 100mm full-flavored and lights prototypes using blend components and flavor systems which will result in a natural blended product.
- B. Status: Blend "C" run at Stockton Street week of October 24. Limited model (1500#) will be run at Semi-works week of November 1 for POL test.
- C. Plans: Analytical and subjective evaluations of "Limited" model.

IV. FLAVOR RESOURCE DATA BANK

- A. Objective: Creation, customization and maintenance of flavor resource and data files for use by Flavor Development Division.
- B. Status: Three new vendor samples have been subjectively evaluated. Aromatic chemical file updated. Lotus 1-2-3 received.
- C. Plans: Division updated with information.

V. INTERNATIONAL SUPPORT

International Brands Smoking Panel

- A. Objective: Subjective evaluations (rod aroma and smoking characteristics) of cigarette brands in the international market.
- B. Status: Panel discontinued during reporting period.
- C. Plans: Provide assistance as needed.

PROJECT LIGHT/ULTRA

- A. Objective: Develop 7mg product for Japanese market.
- B. Status: Awaiting results of P.M.I. testing. Flavor transmittals in progress.
- C. Plans: Factory start-up anticipated for November.

PROJECT MOUNT ULTRA-JAPAN

- A. Objective: Explore the use of new blends, new flavor systems and different construction styles. Cigarettes will be in the low and ultra-low category for the Japanese market.
- B. Status: Awaiting Danchi test results.

PROJECT AVALON

- A. Objective: Development of casing and aftercut systems for a regular and mentholated product for the Asian market.
- B. Status: Models using two blends, two casing systems, eight aftercuts and two menthol application methods have been made.
- C. Plans: Evaluations of prototypes in progress.

PROJECT STARSHIP II

- A. Objective: Produce a 12mg cigarette for the Japanese market.
- B. Status: Awaiting Danchi results.

MEXICO

- A. Objective: Modifications of existing flavor systems used.
- B. Status: Flavor modifications implemented in Marlboro. Additional flavor modification will be evaluated.

PROJECT OLYMPIC (KOREA)

- A. Objective: Development of a product to be competitive to Pine Tree King Size.
- B. Status: Blend has been designed by Blend Development for flavor evaluations. Casings and aftercuts under development.
- C. Plans: Development of casing and aftercut system.

MARLBORO IMPROVEMENT PROGRAM (AUSTRALIA)

- A. Objective: Develop an Australian Marlboro subjectively closer to the U.S. Marlboro.
- B. Status: Blends A and C chosen for laydown in Australia.
- C. Plans: Evaluations of prototypes..

PROJECT NUMBER: 2307
PROJECT TITLE: Flavor Investigation/Nonvolatile Flavor
Investigation/Processed Tobacco
PROJECT LEADER: W. R. Raymond
PERIOD COVERED: October, 1988

I. FLAVOR INVESTIGATION:

A. Objective: To provide analytical support for activities related to development and application of flavoring materials.

B. Results:

1. Project ART: A study was conducted to determine subjective effects of "wax" removal, redistribution and impregnation on extracted ART filler. Stripped, resprayed and impregnated (steam treated and vacuum infiltrated) filler samples were evaluated in hand-made cigarettes against a control using filler exposed to stripping solvent vapor. The control was judged more acceptable than any of the three tests. However, test sample O.V.s were all unacceptably low. Testing will continue with properly equilibrated samples.

ART casing 7983-156 has been scaled up successfully to five gallon batches using the Semi-works 10 gallon casing kettle with modifications. Further scale-up to 30-50 gallons using a larger kettle is in progress. Quantitative methods for residual sugars and glutamic acid have been developed and are in routine use to monitor the casing concentrate for correct degree of processing and the finished casing for correct dilution.

Control Marlboro and a test cigarettes containing RL's with post-ART CRS substituted for 1/6 of Bright stem, prepared small scale in Semi-Works, were analytically and subjectively acceptable and released to PED for internal testing. A large-scale remake of the same control and test cigarettes is in progress in Semi-Works for POL 3609.

2. Analytical Support: Analytical support was provided for ART, Marlboro Reformulation, Half-Pint, Low Density Rod, Natural, and International Product Development. Casing and A/C flavor analyses included: Glycerin/Sugars, 112; PG, 188; EtOH, 24; Water, 16; A/C (anethole and vanillin), 20; Glycyrrhizic Acid, 10; and A/C on Filler, 45. Two ART A/C flavor systems and their components were quantitatively GC profiled and individual constituents identified by GCMS. Complete flavor, casing and filler analyses were run for Marlboro Reformulation and Half-Pint POL production runs at Stockton Street, and ART-stem POL runs in Semi-Works.

Analytical specifications for formula transmittal were generated for Famous (A/C) and Merit Lights, Japan (casing and A/C).

Thirteen flavor chemicals were assayed for purity by GC and impurities identified by GCMS. Questionable purity of mint furanone was investigated and found to be based on quantification of only one of two isomers present. Identification and quantification of the second isomer confirmed specified purity. Isomer ratio found was confirmed as correct by vendor. Twenty-four vendor flavor submissions were profiled by GC and components identified by GCMS for certification and future specifications development.

II. PROCESSED TOBACCO:

A. Objective: To develop basic and applied knowledge for the purpose of improvement or selective modification of subjective properties of processed tobaccos.

B. Results:

Discussions and sample evaluation continued with an alternate vendor of roasted JONEX to move their material toward parity with qualified material from the primary vendor. Analytically, encouraging progress has been made although parity still has not been reached. Initial discussions also have been held with a second alternate vendor and materials will be evaluated as received.

PROJECT NUMBER: 4009
PROJECT TITLE: Smoke Studies
PROJECT LEADER: B. L. Goodman
PERIOD COVERED: October, 1988

PROJECT STUDIO

A. Objective: Develop subjectively acceptable cigarettes with reduced sidestream visibility.

B. Results:

23 mm circumference cigarettes were made from sample bobbins received from Kimberly-Clark to determine the effect of variation in processing of chemical additive levels during their production of the special CaCO_3 paper. The target level of succinate (5 %) and $\pm 15\%$ had been applied by K-C for evaluation of mainstream deliveries and sidestream visibilities. Subjectively, the Studio panelists could tell the difference between the highest and the lowest level, but the variation was too small to significantly affect the acceptability. There was no measurable difference in sidestream visibility nor in mainstream deliveries between the 4.1 % and 5.5 % succinate additive levels. Other K-C papers with an equal level of succinate or citrate were not different from each other in visibility or subjective preference, but the citrate model gave higher mainstream deliveries. Two different types of CMC were also evaluated. There were definite differences between their two models in terms of subjectives. Further samples have been prepared by K-C to determine the best combination of additives on the low sidestream paper.

A cigarette giving maximum sidestream visibility reduction (75%) was achieved by using a perforated, high level of $\text{Mg}(\text{OH})_2$ paper (35%) from Ecusta in combination with a slow burning, porous, thin innerliner from DeMauduit. Other attempts to further decrease the SS smoke, such as using a slower burning blend with DAP on the filler, coarser cuts of the tobacco, or 100% expanded tobacco did not show additional visibility reductions. Analytical data showed that the double wrapped model gave one additional puff over the same paper without the innerliner. Mainstream deliveries were very high for all the models with the slow burning $\text{Mg}(\text{OH})_2$ paper with the exception of the 100% DIET cigarette. The double wrapped model was found to be acceptable subjectively at 1.3 mg tar per puff, but needs to be further designed due to its extremely high puff count. A faster burning $\text{Mg}(\text{OH})_2$ paper gave more normal deliveries and has been requested in a double wrapped configuration.

C. Plans: Subjectively evaluate samples made with a matrix of paper additives on K-C's base sheet for the optimum paper to be used on a 23 mm circumference cigarette.

Combine maximum sidestream visibility reduction from double wrapping Ecusta's different $\text{Mg}(\text{OH})_2$ papers and determine the most desirable mainstream delivery targets.

PROJECT NUMBER: 2304, 2305, 4015
PROJECT TITLE: New Product Development
PROJECT LEADER: B. G. Taylor, H. Maxwell, J. B. Easley, and L. S. Wu
PERIOD COVERED: October, 1988

I. MARLBORO ULTRA LIGHTS

- A. Objective: To develop 85/100 mm Ultra Low (6 mg) candidates for Marlboro line extensions.
- B. Status: POL 4444 (MUL 85mm vs. West German Berlin Marlboro Lights 85mm) results indicated that the Winston Lights 85mm smokers rated the Marlboro Lights higher in liking and preferred it compared to the MUL. The Merit, Vantage and Marlboro Lights smokers showed no significant differences in liking and preference.

POL 4445 (Half Pint 85mm vs. West German Berlin Marlboro Lights 85mm) results indicated that the Merit smokers preferred Half Pint and rated it higher in liking, while the Vantage smokers preferred German Berlin Marlboro Lights and rated it higher in liking. The Winston Lights smokers had no significant difference in liking or preference. There were too few Marlboro Light smokers to report results.

POL 4443 (MUL 85mm vs. Philip Morris Lights American 85mm) and POL 4442 (Half Pint 85mm vs. Philip Morris Lights American 85mm) are on hold waiting panelists.

Models to evaluate blend reformulations were produced in both the 85mm and 100mm designs for Internal MC Testing. The 85mm test was LDB-2 vs. LDB-2R and no significant differences were found. The 100mm test was LDB-2R and a modification of the revised blend (25% BLDET) for greater latitude in puff count control. The modified blend was found to have higher ratings for added flavor and off-taste.

Currently being smoked on the MC Panel is LDB-2R, LDB-3 and LDB-3 with modified flavors in the 85mm configuration. Results should be available in mid-November.

POL 4448 (LDB-2R vs. LDB-3 blends in 85mm configuration) is ready to be overtipped.

C. Plans:

Phase IV POL Tests	4th Qtr., 88
Ad/Pack Testing	4th Qtr., '88
Test Market	1st Qtr., '89

II. Virginia Slims K.S.

- A. Objective: To develop Virginia Slims 85mm products in both conventional and reduced sidestream versions, at 23.0 and 20.0mm circumferences.
- B. Status: The major emphasis is now on the reduced sidestream models at 23.0mm circumference. The blends currently under evaluation include the Virginia Slims blend and the Trim IV blend. Flavor development and evaluation is currently underway. Several low sidestream cigarette papers are currently being evaluated.
- C. Plans:
- | | |
|---------------------------------|----------|
| Prototype Production/Evaluation | On-going |
| Flavor Development/Evaluation | On-going |

III. PROJECT NATURAL

- A. Objective:
- Phase I - To develop "All Natural" cigarette candidates with no additives to the tobacco.
- B. Status: Another production run took place at Stockton Street during October utilizing blend "C". Regular and menthol cigarettes were produced and will undergo spotting evaluations. Eight more paper samples have been received from Ecusta and will be evaluated in Semiworks during November.
- C. Plans:
- | | |
|----------------------------|----------|
| Study Anti-Spotting Papers | On-going |
| Menthol | On-going |

IV. PROJECT EXTRA

- A. Objective: The development of 4-8 mg product candidates that have the taste of products with twice the tar.
- B. Status: The designs have been completed to test the theory of lower dilution due to lower rod RTD for low density products. The cigarettes were made 10/31/88 and are in analytical.

Analytical data has been completed on the Aftercut Application study. The results indicate that the most efficient application of aftercut was via post-pectin-coating. The least effective method was to apply the aftercut during pectin-coating. As a result of the findings, the All-lamina blend will be produced with 8493-188A casing and Marlboro aftercut at various levels.

Subjective results of cigarettes produced via the Batch process vs. the Continuous process show no significant differences.

A program to develop a 6mg free-standing cigarette which appeals to flavor low smokers was initiated. Three blends, recommended by the Leaf Department, have been evaluated with one chosen for Aftercut work with both CA and paper/CA filters. Four models have been requested from which a candidate will be chosen to test on POL vs. Half Pint.

C. Plans:

Evaluate paper/ca filter variability	4th Qtr. '88
Evaluate alternate filters	On-going
Puff profile	On-going
Low density rod program	On-going

V. Project 202

A. Objective: To develop a 2-3mg "Merit-type" cigarette.

B. Status: This is a spin-off of the Extra Project.

Modified Half Pint blend with 25% expanded, Half Pint casings and AC, paper/CA dual filter has been selected for the Saudi market. Specifications are being written.

Further work in this area for the U.S. market will continue under Project Merit Super Lights. Aftercuts are being evaluated on two blend models. Both blends have been cased with Total blend casing 7695-163D.

C. Plans:

Specifications	11/88
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VI. PROJECT VALUE ENTRY

A. Objective: To develop products to compete in the Value Entry Categories

B. Status:

Alpine

The Generic Alpine brand family has done well in Test Market and the plans are for National introduction early January 1989. The product for the National Introduction will have a lower cost modified MB4 blend.

Target

POL 4439 (Cambridge Lights 85mm vs Target Model II 85mm) results showed no significant differences between the cigarette ratings; however, the liking ratings were (4.79 to 4.97) indicating the Target would be acceptable among Winston Lights, Century Lights, and Doral Lights smokers.

POL 3592 (Cambridge Full Flavor 85mm vs Target Model II 85mm) was requested to determine consumer acceptability of the new Target blend and flavor system by Winston and other RJR smokers. The test results showed no significant differences between the cigarette ratings for the Winston smokers. The Doral smokers preferred Cambridge and rated Target higher on cool smoking. The Century smokers rated Cambridge higher on harshness and Target higher on mild taste. The liking ratings were (4.79 to 5.30) indicating the Target would be acceptable among Winston, Century, and Doral smokers.

POL 3608 (Target Model II 85mm vs Winston 85mm) was requested to determine the consumer acceptability of the new Target blend and flavor system by Winston and other RJR smokers in comparison with Winston. Currently awaiting cigarette make for the Target Model II 85mm.

C. Plans:

Alpine National Introduction	1/89
Target Ad/Pack Testing/Factory Trial	On-hold

VII. B&H KING SIZE

- A. **Objective:** To develop a B&H 83mm box product with comparable subjectives of B&H 100 in both a full flavor and lights version, both nonmenthol and menthol.
- B. **Status:** A series of Ad-Pack and Consumer Testings Studies have been requested by New York. The testing involves six different models: two blends - B&H and Natural Blend C; Regular and Menthol; and full flavor and lights. The nonmenthol cigarettes were made, packed and shipped to New York. The menthol cigarettes will be made when packaging material is available.

C. Plans:

Menthol Ad/Pack Cigarettes Produced	11/88
Blend and flavor work	On-going
Test Market	1st Qtr. '89

VIII. Marlboro Blend Reformulation

- A. Objectives: To remove off shore tobacco from Marlboro blend.
- B. Status: Phase XI cigarettes were produced in early October. This incorporated blends 11 and 11-1. These cigarettes were acceptable analytically and subjectively and will be shipped to panelists the first week in November. A repeat of Phase XI will be produced at Stockton Street in early November.

C. Plans:

Phase XI repeat

11/88

PROJECT NUMBER: 4022
PROJECT TITLE: International Product Development
PERIOD COVERED: October, 1988
WRITTEN BY: A. H. Confer

I. PROJECT DRAGON (PEOPLES REPUBLIC OF CHINA)

- A. Objective: Develop a "blended" KS cigarette to be a non-PM trademark owned and manufactured by CNTC. Tar targets: 17-19 mg. (GCF-2 factory), and less than 15 mg. (ZCF factory).
- B. Results: A book summarizing blends, casings/flavors, and cigarette construction for the proposed prototypes at ZCF was completed and sent to Hong Kong for translation. Sample quantities of the necessary leaf grades and casings/flavors were sent to ZCF.
- C. Plans: A Chinese delegation will visit Richmond, November 15-18. At that time Project Dragon will be reviewed, and prototypes subjectively evaluated.

II. MARLBORO IMPROVEMENT (AUSTRALIA)

- A. Objective: Modify the Australian Marlboro to bring it subjectively closer to the USA product.
- B. Results: Tobacco grades and casings/flavors were prepared and shipped to Australia for prototype production.
- C. Plans: Two 3,000 kg. test blends are tentatively scheduled to be run during the week of November 7.

III. PROJECT FAMOUS (WORLDWIDE)

- A. Objective: Optimize Chesterfield as a worldwide competitor to Camel.
- B. Results: POL 3589 (Famous 2A vs. Camel) was completed. Among Marlboro smokers, Camel was directionally preferred over Famous 2A. Among Camel smokers, Famous 2A was directionally preferred over Camel.
- C. Plans: POL 3593 (Famous 2B vs. Camel) will close out November 7.

PROJECT NUMBER: 4024
PROJECT TITLE: Japan Product Development
PERIOD COVERED: October, 1988
WRITTEN BY: J. N. Smith

I. MERIT LIGHTS

- A. Objective: Development of a 7 mg tar product to compete with Mild Seven Lights and other mainstream Japanese smokers.
- B. Results: Results of the PMI testing indicate that there was no statistical difference in preference between the two white tipped Merit Lights models and Mild Seven Lights among a cross section of Japanese smokers.
- C. Plans: A factory trial to finalize the product specifications is scheduled for November 8 at the M/C. Production start-up is scheduled for mid-November. A U.S. testing certification is planned for the initial production run.

II. CHESTERFIELD

- A. Objective: Develop an 11 to 12 mg American blended product.
- B. Results: In the second Danchi test, the Model II prototype again received the highest liking score of the products tested.
- C. Plans: Results of the PMI test versus Lucky Strike are expected by mid-November.

III. MARLBORO

- A. Objective: Improvement to the subjective response of the Marlboro Full Flavor product.
- B. Results: In the Marlboro Matrix testing, the New Marlboro was significantly preferred over the other three products tested. These results are similar to previously reported Danchi panel results.
- C. Plans: Marlboro KS and 100's specifications are being prepared for discussions with JTI.

IV. MOMENT

- A. Objective: Development of a menthol product acceptable to the Japanese consumer.

- B. **Results**: One Danchi has been shipped this month which will examine blend, tar level, and menthol level.
- C. **Plans**: A second Danchi is being prepared for shipment in December which is designed to test additional tar and menthol levels.

PROJECT NUMBER: 5001
PROJECT TITLE: Packaging Studies
PROJECT LEADER: H. R. Dunaway
PERIOD COVERED: October, 1988

PACKAGING STUDIES

A. Objective: Provide technical packaging support to Manufacturing, Manufacturing Services, Engineering, Purchasing, and Quality Assurance. In addition, assist New Products Directorate in evaluating new packaging concepts and products.

B. Status/Plans:

Residual Printing Solvents in Packaging Materials: Evaluation of the following items was coordinated in the past month.

- Marlboro KS SP Foil Carton Overwrap - General Foods
- Alpine Singapore Ink Samples - JWF
- B&H 100 FF SP 20 - IPC
- Merit UL KS SP 20 - GB
- PALL MALL RED LABEL - GB
- PALL MALL GOLD LABEL - GB
- CARLTON LTS BLUE LABEL - GB
- Diisononyl phthalate - Strahan Ink
- Marl FT 20 (Ivory Board) from Indonesia
- B&H 100 FF SP 20 - Alford
- 5 SP Labels With Experimental Ink Systems - JWF
- Alpine LTS 100 SP 20 - IPC
- Alpine FF 100 SP 20 - IPC
- BUCKS KS SP 20 - Alford
- BUCKS 100 SP 20 - Alford
- Merit Carton Inserts - Weber
- Virginia Slims Carton Inserts - Weber
- Marl Men LTS SP Carton Insert - AGI
- Gridlock NonFilter KS SP 20/200 - GP
- Gridlock NonFilter KS SP 20 - JWF
- Sample gravure printed labels - JWF
- FVB Non-Filter KS SP 20/200 - SP
- FVB Non-Filter KS SP 20 - IPC

Purchasing was advised on the acceptability of the above items for use in production, and where necessary, recommendations were made on the proper handling prior to use.

Poly Laminated Blanks and Cartons: A trial run was made at semi-works with two possible glue candidates for use on SBS board with polypropylene laminated to the inside surface. A short environmental test was conducted to determine if either of the adhesives failed under adverse conditions. An adhesive has been chosen, and a 12 week full case OV study is planned to evaluate these materials for improved moisture retention.

PROJECT NUMBER: 0400
PROJECT TITLE: Low Density Rod Development
PROJECT LEADER: R. S. Mullins
PERIOD COVERED: October 1988

LOW DENSITY ROD

- A. Objective: Develop a continuous process for the production of reduced density cigarettes.
- B. Results: Analytical results from the aftercut application/tracking study indicated that approximately one third of the anethole was lost during subsequent processing when the aftercut was applied to the all-lamina blend in the small scale primary either before or after the binder. When the aftercut was applied before the binder, most of the loss occurred during binder application, with no loss detected during cigarette making. When the aftercut was applied after the binder, the loss occurred during filler storage and cigarette making. When the aftercut was applied with the binder, only trace levels of anethole could be detected in either the filler or the cigarettes. Subjective evaluation of the samples is in progress but is being slowed by the ART program. A follow-up test is in progress to confirm the losses using Marlboro filler with the aftercut applied in the large scale primary.

A series of low density cigarette models has been produced for a test to determine if reduced cigarette density allows a significant reduction in dilution, at constant delivery, puff count, and RTD. Analytical testing of the samples will begin following packing of the cigarettes during the second week of November.

Production plans for the second set of samples for Project Tomorrow have been finalized. Binder will be applied to 20 cpi Marlboro without aftercut in the continuous coating cylinder. Following binder application, aftercut will be applied to the filler in the Semiworks, at an increased rate to account for losses during cigarette making. Untipped, 63 mm cigarette rods will be produced and, after weight selection, tipped and packed in the Semiworks. Following analytical testing and subjective screening by an expert panel, the cigarettes will be subjectively evaluated by the M/C panel.

Firmness and loose ends results from the circumference variation study provided no indication of increased bond or filler breakage at reduced circumferences. Analysis of data from the tipper degradation test is underway.

A test to determine if rejected bound cigarettes can be recovered using the standard ripping process was conducted at Stockton Street. No problems were encountered in ripping the bound cigarettes and no unusual build-up of material in the equipment was noticed during the test. Samples collected throughout the run are being tested by Stockton Street QA personnel. During production of the 550,000 low density cigarettes needed for this test, no significant problems were encountered in operating the cigarette maker at speeds as high as 1800 cpm.

- C. **Plans:** Produce cigarette samples for Project Tomorrow in a Marlboro configuration. Complete packing and testing of the cigarettes produced for the dilution reduction study. Evaluate the results from the circumference variation and tipper tests. Evaluate the results of the test conducted to determine if rejected low density cigarettes can be recovered using the standard factory ripper process. Evaluate moisture application to the filler in the chimney using an ultrasonic nozzle.

PROJECT NUMBER: 1307
PROJECT TITLE: Reconstituted Tobacco Development
PROJECT LEADER: R. G. Uhl
PERIOD COVERED: October, 1988

I. IMPROVED SHEET PROPERTIES

A. Objective: Improve the physical characteristics and blend properties of reconstituted sheet materials.

B. Results:

1. ART Project - The POL cigarettes produced to evaluate ART stem inclusion (half utilization level) in pilot RL were rejected for low aftercut flavor. Blend remakes are in progress in Semiworks.

Small-scale cigarettes containing pilot RL which incorporates both half and full utilization levels of ART stem are being subjectively evaluated by PED on the MC internal panel. Results will be available in November.

RCB handsheets made with several levels of ART stems have not been screened due to competing ART subjective priorities.

2. Humectants - Glycerine-free RCB handsheets were made with several levels of supplemental sugars (isosweet and sucrose) to determine if this would improve the subjective properties. The subjective shortfall in previous test products appears more related to the absence of class tobacco casings when CT feedstock is replaced by stemmery scrap to produce a glycerine-free RCB, than it is to the removal of glycerine. These sheets will be screened in handmade 100% cigarettes.

Semiworks survivability testing was completed on pilot RL sheets containing individual humectants (4% glycerine, 4% PG, 4% isosweet, or no humectant). Combined longs plus mediums exit the maker garniture showed no effect due to cutter OV, but had a strong direct relationship to maker OV. Piece size versus maker OV plots for three of the sheets (glycerine, isosweet, no humectant) were superimposed, indicating that moisture content alone governs RL survivability. The PG sheet showed lower survivability on an equal OV basis; correcting OV results for PG volatility in the OV test gave the PG sheet at least equal survivability on a constant moisture basis.

3. RL - Pilot RL was produced incorporating fatty acids in the size formulation for the Microbiology Group to determine the subjective acceptability of these materials. Fatty acids have shown promise as SEL preservatives in laboratory incubation studies.
4. Other Recon - Samples of Philippine Heiboflake sheets made using alternate formulations were evaluated on the small-scale

impact tester. Three of the sheets, made with higher binder and/or humectant contents, showed some improvement in survivability.

C. Plans:

1. Provide RL pilot plant support for the POL evaluation of ART stems in RL.
2. Expedite subjective evaluation of ART stems in RCB handsheets.
3. Expedite subjective evaluation of glycerine-free RCB handsheets made with additional sugars.

II. SUBJECTIVE MODIFICATION OF RL

A. Objective: Improve or modify the subjective character of RL.

B. Results:

1. Dry Flavor Replacement - Flavor Development is continuing the analytical evaluation of vendor native extracts of liquid flavors following vendor modifications of the roasting process. These flavors will be evaluated in pilot RL when they become available.
2. Modified 150B - The Branson ultrasound processing unit was installed and tested. Modified 150B trials will be commence in late November

C. Plans:

1. Produce pilot RL to evaluate liquid flavors as available.
2. Commence trials to determine if ultrasound will break up Modified 150B size precipitates.

III. CIGARETTE PAPER DEVELOPMENT

A. Objective: Support development of proprietary low sidestream cigarette papers.

B. Results:

1. Handsheets - Beater refining curves were developed for the available flax fiber. Refining was continued to the extreme in order to achieve the very low paper porosity target. Porosities in the desired range were achieved in handsheets, using greater press section compaction than available in Maine. Freeness, a measurement of the degree of stock refining, loses its utility with heavily refined stock; freeness changes only slightly as refining is continued, while the resulting paper shows progressively lower porosity. This

will make the degree of refining difficult to control in a pilot or production situation.

2. Pilot Trials - The rolls of test cigarette paper produced at the University of Maine trials were slit into 72mm wide bobbins at Colonial Heights Packaging to allow sizing on the Product Development mini-press. A second set of trials has been scheduled for the University of Maine in November. Sheet production will be targeted for a Coresta porosity of 5.5 using a 40 gm/m² sheet and the finer (0.07 micron) carbonate filler. The use of a disc refiner as a means of rapid final freeness adjustment will also be evaluated.

C. Plans:

1. Evaluate alternate (non-flax) fiber types as a means of improving cigarette paper properties.
2. Conduct a second set of trials at the University of Maine to produce bobbins of low porosity papers for evaluation.

PROJECT NUMBER: 1333
PROJECT TITLE: Semiworks Process Control
PROJECT LEADER: D. A. Phan
PERIOD COVERED: October, 1988

- A. Objective: Evaluate and revise the process control and data acquisition system to improve processing performance and production quality.

B. Results:

Hauni HT Steam Tunnel Installation (Oliver) - Continue to support Dr. Cho's program to evaluate the Hauni steam tunnel. Modifications to the weighbelt at the inlet of the tunnel will be performed to increase its speed and reduce tobacco bed depth. This is to provide better tobacco feed to the tunnel. Other instruments will be installed to monitor the spray steam temperature and pressure, and tobacco temperature at the discharge of the tunnel and the inlet of the Adt dryer.

Temperature Control System for Scandia Overwrappers (Osmalov/Phan/Sims) A temperature control system and most of the mechanical modifications were installed on one of the Scandia overwrappers at the Stockton St. plant on 10/8. Other mechanical modifications are planned to be done by 11/15. Pack-seal quality improvement will then be evaluated by factory personnel.

Second Aftercut Flavor Cylinder (Phan/Sims) - The Fenwal fire and explosion suppression system for the cylinder was installed and checked out. Electrical and control installation for the flavor system and associated conveyors began on 11/7. The system will be checked out the week of 11/14.

Vacuum Conditioner Data Highway Connection (Sims) - Work is underway to connect a Provox data highway to the stand-alone Provox system at the vacuum conditioner panel. This will allow better maintenance control of software from the central system, and opportunity of trending some important process variables at that location. The project is planned to be completed by November 30, 1988.

Automatic Damper System for the Adt Dryer (Sims) - Work is underway to provide an automatic system to allow remote switching of the inlet and exhaust air dampers from the control room for co-current or counter-current air flow modes. The project is planned to be completed by December 15, 1988.

- C. Plans - Complete the checkout of the second aftercut flavor system, and the data highway for the vacuum conditioner control system. Complete the installation of all remaining items to support the Hauni steam tunnel evaluation program. Continue the development of the Onspec control software and qualification of the Computrac moisture analyzers. Continue providing electrical plant engineering support to the Semiworks and conduct routine QA functions.

PROJECT NUMBER:: 1503
PROJECT TITLE:: Modified Smoking Materials
PROJECT LEADER:: W. A. Nichols
PERIOD COVERED:: October, 1988

I. LOW DENSITY ROD - BINDER APPLICATION

A. **Objective:** Produce precoated filler via the batch process to support the current product and process development efforts on the low density rod program. Develop a method of pre-applying binders to the tobacco in a manner that can be scaled up to commercial scale.

B. **Results:** Experimentation conducted with pectin solutions from 2% to 8% (6% add-on) has demonstrated that cylinder buildup is primarily a function of solution flow rate and is insensitive to viscosity.

Results from studies comparing the batch and continuous coating processes indicate no difference in subjectives or physical properties. Additional testing will be performed to further verify this conclusion.

Process runs on the coating reel indicate that output can be increased from 60 lb/hr to 80 lb/hr without problems in coating.

C. **Plans:** Tracer investigations will be completed and standardized testing established. Tests will be performed to determine the optimum coated filler OV for cigarette fabrication.

II. ADDITIVE APPLICATION PROCESS

A. **Objective:** Utilize foam bound rod fluid application technology and other technologies for the application of cigarette additives.

B. **Results:** Sample cigarettes were produced to Marlboro Lights specification with 3.7, 3.9, 4.1, and 4.3 mg menthol/cigt. with the Kaymich applicator. Aging studies and subjective testing will be conducted on the samples.

Several issues pertaining to implementation of the Kaymich system into Manufacturing were discussed with Operations personnel. Menthol losses in the Oscar, handling of rejects, contamination, etc., will be examined in future testing.

C. **Plans:** Samples will be produced with higher GMC loading to determine if spots can be decreased. Flowmeters in the Kaymich Applicator will be tested for viscosity sensitivity. Product testing of menthol samples will be completed.

PROJECT NUMBER: 1806
PROJECT TITLE: New Tobacco Processes
SECTION LEADER: S. R. Wagoner
PERIOD COVERED: October, 1988

I. PROJECT ART - COMMERCIAL PROCESS DEVELOPMENT

- A. Objective: To conduct trials providing information for development of the ART commercial process.
- B. Results: Spotting analyses were conducted on several sets of cigarettes containing unextracted and extracted ART filler. The following results were observed:
1. Tests on extracted filler from the one liter laboratory did not show the tendency for spotting that has been observed in pilot plant material.
 2. From an inspection of Bermuda Hundred Pilot Plant runs 220 and 221, it was concluded that extracted filler from the wax layer created significantly more spots than filler from the main body of the bed.
 3. Cigarettes made from unextracted DL blend, and subjected to a standard QA defect audit, yielded 0.7 critical spots per 2000 cigarettes. (The third quarter 1988 factory audit showed 1.34 critical spots/2000 cig.)
 4. Cigarettes from ART run 226, in which the filler was cased and dried immediately after extraction, yielded 12.5 critical spots/2000 cig in the standard QA audit. In addition, the QA audit of runs 238/239, which were precased with PG and glycerin in the AB solution, showed 29.2 critical spots/2000 cig.
 5. A similar QA inspection of cigarettes containing 100% expanded ART filler yielded 12.0 critical spots/2000 cig.
 6. Quality Engineering utilized the off-line inspection device to evaluate several sets of the above cigarettes. Their results showed that the unextracted DL filler produced 13.0 critical spots/2000 cig and the PG/G precased ART filler produced 45.5 critical spots/2000 cig. (For the QE device, Marlboro typically runs from 8 to 25 critical spots/2000 cigs.)

Samples of unextracted and extracted ART fillers were submitted to analytical microscopy for characterization of the surface. The unextracted filler was clean with no extra waxy deposits on the epidermal surface. Post-ART filler was shown to be covered with a combination of waxy substances. In the laboratory, various ART samples were dried in the vacuum oven. This treatment significantly reduced the amount of spots produced in the accelerated spotting test.

A CV/OV/RH regression analysis of ART filler was conducted. Unextracted DL3 filler, uncased post-ART filler (run 207), and post-ART filler with casing and after-cut were conditioned at three humidity levels and analyzed for equilibrated CV/OV. Correction factors of 0.44, 0.45, and 0.41 cc/g/%OV, respectively, were generated for the above fillers.

Expansion runs of ART filler were conducted for Product Development work. All trials were run immediately following extraction, and used a tower temperature of 500F.

A list of acids and the corrosion potentials on Inconel and stainless steel was received from Bernie LaRoy to determine replacement levels for 12% monopotassium citrate on stem. Dissociation constants were found in the literature and relationships with temperature were determined. The ART process computer model was used to calculate the appropriate add-on levels.

- C. **Plans:** With the aid of QA and QE, continue to quantify the level of cigarette spotting caused by ART filler. Conduct post-ART tobacco treatment tests with the Hauni HT tunnel and the vacuum conditioner to determine the effect on spotting.

II. BINDER DEVELOPMENT

- A. **Objective:** Develop methods to produce binder systems for the foam bonded ends and low density rod programs.
- B. **Results:** For the foam bonded ends program, CMC solutions of sodium, potassium, magnesium, and calcium salts were used to tip the ends of cigarettes. These will be subjectively evaluated for flavor enhancement of CMC.

A meeting was held with technical representatives from Grindsted to discuss the physical properties of pectin and their quantification. Surface tension and contact angle measurements were recommended, and our lab instruments will be modified to conduct these analyses for tobacco coated with pectin. Discussions included the use of emulsifiers to enhance adhesive quality, and a sample of the tartaric ester will be sent to us for experimentation.

- C. **Plans:** Continue to provide support as required to the above programs.

III. HAUNI HT TUNNEL EVALUATION

- A. **Objective:** Determine the effect of steam conditioning cut filler prior to drying in the Hauni HT steam tunnel.
- B. **Results:** Check-out tests, using Marlboro filler exit production cutters, were conducted on the newly installed Hauni HT tunnel in

the Semiworks. A punch list of modifications is being addressed. Equilibrated CV data have shown a 0.4-0.6 cc/g increase across the tunnel in these preliminary trials.

- C. Plans: Complete equipment modifications such that an evaluation program can be conducted to determine the physical, chemical, and subjective characteristics of steam conditioned cut filler.

IV. TMCI-ASTA SHEET

- A. Objective: To develop a subjectively and physically acceptable reconstituted tobacco sheet using the TMCI process and PM-RCB technology for international application.
- B. Results: The Spanish tobacco to be used for particle size optimization trials in Tarragona was ground in Nuway Tobacco Company. Three grinds were made: (a) 90% <400 mesh, mean particle size (mps) 28 μ ; (b) 95% <200 mesh, mps 42 μ ; (c) 95% <120 mesh, mps 55 μ . (the normal grind in Tarragona is 65% <120 mesh, mps 104 μ). Sufficient quantities of each grind (3000 lbs) were made to allow 8 hours operation at design throughput. The tobacco was passed by USDA for insecticide content on 10/6 and was sent to Spain after grinding on 10/18. The tobacco is now in Tarragona. The total reducing sugar content of this Spanish blend is again high (8.6% DWB) and no additional sugar will be required.

The slits in the ASTA product made during the May trials using fine dust (95% <400 mesh) were reproduced in the R&D laboratory. Water droplets were projected in an air stream across a partially formed handsheet. The slits were identical to those in the Tarragona product confirming that these slits were made by steam condensate escaping from the underside of the belt through the broken sealing bars.

C. Plans:

Evaluate the mechanical modifications to improve the Tarragona plant operation by running trials using tobacco ground in Nuway to 95% <400 mesh).

Witness one week of satisfactory ASTA process operation using the normal tobacco grind before running trials to optimize tobacco particle size in Tarragona.

PROJECT NUMBER: 1810
PROJECT TITLE: Project ART
PROJECT LEADER: Ravi Prasad
PERIOD COVERED: October, 1988

I. PROJECT ART

- A. Objective: To support commercial Plant Design and Flavor Development objectives at the Bermuda Hundred Pilot Plant.
- B. Results: Pilot Plant tests with the fourth master batch of DL blend (DL-4) indicated that the extractability of this batch is similar to the earlier batches, i.e., 97% nicotine reduction can be achieved at standard process conditions.

Stem absorber optimization indicated that the absorber pressure drop can be reduced by reducing the CRS cuts-per-inch. However, the nicotine absorption efficiency drops with the use of reduced CPI stems. Further optimization activity has been deferred, as the priority was shifted toward Flavor Development objectives.

Preferred process conditions were selected for pilot plant operation to best support the Flavor Development activity, i.e., reduced spotting with cold recovery of CO₂, 97% nicotine removal. Addition of humectants prior to extraction was also implemented to improve the product quality. To-date 97% nicotine reduction has been achieved in all runs that did not have mechanical malfunction, i.e., CO₂ leaks or power failure.

In support of the Commercial Plant, nicotine laden activated carbon (950 lbs) was thermally treated to determine the scale-up of laboratory thermal treatment procedures.

Liquid samples from the extraction room were taken to determine the potential for metal corrosion/pitting. Data on "chloride" content in the system was provided to determine its impact on corrosion for both the Pilot Plant and the Commercial Plant.

Ongoing support to the Analytical Division was continued via the analysis of key samples to determine the variability in nicotine measurement. In addition, an internal check of the Hewlett Packard GC Model 5880A at the pilot plant is being done to assure that nicotine measurements are accurate from day-to-day.

- C. Plans: Continue production in support of Flavor Development activity.

PROJECT NUMBER: 1101
PROJECT TITLE: Entomological Research
PROJECT LEADER: D. L. Faustini
WRITTEN BY: M. F. Minor
PERIOD COVERED: October, 1988

I. PHYSIOLOGICAL STUDIES OF THE CIGARETTE BEETLE (CB)

A. STUDY OF GLOBAL ECOLOGY

1. Objective: To determine the distribution and abundance of the CB as it relates to the processing of tobacco into cigarettes.
2. Results: Pheromone traps were installed at ULT's Richmond, Virginia and Shelbyville, Kentucky hogshead storage/refurbishing facilities on 9-16-88. Monitoring programs at Cabarrus (8-28-88) and Louisville (9-13-88) were reviewed.¹
3. Plans: Continuation of monitoring the traps.

B. STUDY OF FLAVOR EXTRACT

1. Objective: To determine the attraction of certain flavor extracts to female adult cigarette beetles (CBs).
2. Results: Prune extract as obtained from the supplier was tested for attractiveness to 200 mixed-sex CBs in the olfactometer. With more than a 40% increase of CBs in the target chambers as compared with the controls, prune extract was highly attractive. Raisin extract as obtained from the supplier was studied under the same conditions as the prune extract and proved to be unattractive to CBs.²
3. Plans: Continue to study the rest of the flavors. Sex the CBs and test the females with flavors found to be attractive to mixed sex CBs.

C. STUDY OF STEM BIOASSAYS

1. Objective: To determine the optimum methoprene treatment level and storage time of stems and stem scraps.
2. Results: Cigarette beetle emergence is progressing in baseline bioassays.³
3. Plans: Initiate bioassays in treated stems and scraps.

D. REFERENCES

1. Ryan, L. Memo to D. L. Faustini, September 16, 1988.
2. Minor, M. F. Notebook #8539, p. 65.
3. Lehman, R. M. Notebook #8519, pp. 97 - 100.

II. SERVICE TO OTHERS

- A. Objective: To conduct and provide technical services to areas outside R&D.
- B. Results: Insect fragments found in reprocessed strip at the Tobacco Processing Facility (20th St.) were identified.¹ A recommendation on contact (space spray) insecticides was made to Fabriques de Tabac Reunies (Neuchâtel, S.A.) at the Onnens warehouses.²
- C. References:
1. Faustini, D. L. Memo to Frank Pajaczkowski. Insect Fragments in Reprocessed Strip. October 12, 1988.
 2. Faustini, D. L. Memo to Dieter Schulthess. Contact Space Spray Insecticides. October 14, 1988.

PROJECT NUMBER: 1620
PROJECT TITLE: Electrophysiological Studies
PROJECT LEADER: F. P. Gullotta
WRITTEN BY: C. S. Hayes
PERIOD COVERED: October, 1988

I. NASAL EVENT-RELATED POTENTIALS (NERPs)

- A. Objective: To develop methods to objectively and reliably evaluate human responses to cigarettes, smoke constituents and tobacco flavorants.

B. Results:

Cognitive NERP Study

Flavor discrimination experiments comparing natural and synthetic menthol have been completed in ten subjects utilizing the cognitive odd-ball paradigm.¹¹ Both the electrophysiological and the psychophysical data indicate that subjects are good at differentiating between the two different menthols. A late positive component (LPC) was recorded to the natural menthol targets that was not present or extremely diminished to the synthetic standards. As expected, the data revealed that this discrimination was more difficult than the CO₂ vs natural menthol discrimination. That is, stimulus reaction times were longer and the LPCs were smaller and longer in latency to the synthetic/natural menthol discrimination as compared to the CO₂/natural menthol discrimination. Additionally, subjects made more misses (*i.e.*, failed to identify the target stimulus) and false alarms (*i.e.*, responded to a standard stimulus as if it were a target) in the synthetic vs natural menthol discrimination. These data confirm that a relationship exists between the LPC of the NERP and discriminability. Furthermore, they indicate the utility of the LPC technique for not only determining whether individuals can discriminate among flavors, but also the degree of their discriminability.

Statistical analyses aimed at quantifying the differences between target and standard NERPs for both the CO₂ vs menthol and natural vs synthetic menthol comparisons are currently in progress. Analyses are also being conducted on missed targets as well as false alarms. Experiments comparing a mixture of natural and synthetic menthol to 100% synthetic menthol have been initiated in the same ten subjects.

- C. Plans: Complete testing of ten subjects in the cognitive paradigm comparing a mixture of natural and synthetic menthol to 100% synthetic menthol. Continue statistical analyses of the data from both the CO₂ vs natural menthol and the natural vs synthetic menthol comparisons in order to quantify the differences between the responses.
- D. Reference: Martin, B. R. Notebook No. 8689, pp. 85-109.

PROJECT NUMBER: 1702
PROJECT TITLE: Optical Processing and Aerosol Research
PROJECT LEADER: K. A. Cox
PERIOD COVERED: October, 1988

I. PACK INSPECTION (R. Maher and C. Harward)

- A. Objective: Develop and implement a method for digital pack inspection.
- B. Results: After a review of a number of digital imaging systems, the system manufactured by Androx of Canton, MA, has been selected for implementation of the digital inspection algorithm. Androx will supply a single board which, when used with the Sun 3 microcomputer, can execute the inspection algorithm at the rate of 10 per second. Furthermore, the calculations needed in the training step can be carried out on the imaging board, thereby reducing the time required for this step.
- C. Plans: Implement the inspection algorithm using the Androx imaging board. Carry out a systematic evaluation of the performance of the system using a set of both acceptable and defective Lark cigarette packs.

II. AEROSOL RESEARCH (T. Nguyen)

- A. Objective: Add the constraint of non-negativity to the algorithm for obtaining aerosol particle size distributions (PSD) from light extinction data.
- B. Results: Available algorithms for obtaining aerosol particle size distributions from light extinction data often yield unphysical results when the data contains even a small amount of error. Our method for inverting extinction data was modified to incorporate the constraint that the PSD be everywhere positive. The algorithm has been found to work very well when tested with data at a variety of error levels.
- C. Plans: Carry out a systematic evaluation of the capabilities of the algorithm.

III. INDIVIDUAL CIGARETTE INSPECTION (D. Lowitz)

- A. Objective: Develop methods for the online inspection of individual cigarettes.
- B. Results: A meeting was held with Reticon personnel to discuss the possibility of using their linear CCD array for image acquisition in the individual cigarette inspection system. The speed of the array was found to be insufficient and the lighting requirements were expected to be excessive. The acoustooptic linear scanner proposed by Brimose Corporation now appears to be our best choice,

although the applicability of a rotating polygon mirror scanning system remains under consideration.

IV. GLOBAL HOLONETICS "SMART CAMERA" (Charles Harward)

- A. Objective: Evaluate the defect detection capability of the Global Holonetics "Smart Camera" (SC).
- B. Results: The in-house evaluation of the Smart Camera is still on hold due to temperature sensitivity of the device. Global has been working to correct this problem since the middle of June. Global has worked with HNC (the neural net people) to develop an algorithm to distinguish between good and defective packs where the system was trained on only good packs. They have been successful in distinguishing good cigarette packs from cigarette packs with no closure stamps, closure stamps in the wrong place, and packs with side-to-side registration misalignment. They expect to ship us the first prototype system with the neural network option in mid-November.
- C. Plans: Evaluate the inspection system when it becomes available.

PROJECT NUMBER: 1704
PROJECT TITLE: Supercritical Fluid Processes
PROJECT LEADER: T. M. Howell
PERIOD COVERED: October, 1988

I. LOW NICOTINE

- A. Objective: Understand solubility behavior of tobacco wax solubles in SC-CO₂.
- B. Results: Fabrication of a unit that will permit varying pressure and volume while maintaining constant temperature is complete and testing has begun. Filler is equilibrated with SC-CO₂ at standard operating conditions. A portion of the stream is isolated in a Ruska view cell through which the beam from a helium-neon laser is passed and the light scattering is detected. The volume of the cell is expanded to reduce pressure while temperature is held constant. When the cloud point is reached the fluid becomes turbid and the degree of turbidity is measured by the detector. A pressure-temperature relation for precipitation of the solubles can then be obtained. The results using DL4 blend at 12% OV are shown below.

Temperature	CO ₂ :Tob	Pressure @ Prec
60	10.8:1	3150
60	22.2	3000
50	10.8:1	2900
50	22.2:1	2750

The data indicate that the soluble material is in the retrograde region and that at current operating conditions the CO₂ is not saturated with these compounds.

- C. Plans: Continue developing this technique in order to better understand wax deposition and spotting during our current let-down procedures.

II. LOW NICOTINE

- A. Objective: Evaluate errors associated with analysis and tobacco preparation.
- B. Results: A study was carried out within the SCF group to check the accuracy and precision of our nicotine, AB, and OV analyses. Initial evaluation of the data uncovered some areas where improvements could be made.
- C. Plans: Finish evaluating the data and improve where possible. Develop a maintenance program for continued minimization of errors.

III. LOW NICOTINE

- A. Objective: Develop safety protocol for the SCF laboratory.
- B. Results: Installation of a gas monitoring system is near completion. Development of a documented safety protocol was initiated with an evaluation of current procedures.
- C. Plans: Finalize and document safety protocol.

IV. LOW NICOTINE

- A. Objective: Develop alternate on-line nicotine monitors for the ART process.
- B. Results: A shorter path length cell has been received and pressure checked. Installation is underway.
- C. Plans: This work is ongoing.

PROJECT NUMBER: 1708
PROJECT TITLE: Physical Chemistry and Process Monitoring
PROJECT LEADER: J. L. Banyasz
PERIOD COVERED: October, 1988

I. OPERATIONS SUPPORT (J. Crump and A. Closter, in collaboration with the Applied Technology Group)

- A. Objective: Determine the effect of particle size distribution on dynamic viscosity changes that occur in PVA tipping adhesives.
- B. Results: The roller speed study continues. A memo detailing the results to date has been issued.

Work has been initiated on the fabrication of rollers of varying hardnesses. The test rollers will be used to determine whether particle size degradation can be reduced by going to a softer roller.

II. OPERATIONS SUPPORT (P. Henderson, in collaboration with the Applied Technology Group)

- A. Objective: Characterization of inks.
- B. Results: Contact was established with Weber and Permut, Inc., an ink consulting firm, in order to obtain assistance with the standardization of inks and the establishment of quality control criteria.

III. OPERATIONS SUPPORT (S. Ganeriwala)

- A. Objective: Assess the effectiveness of adhesives used in overtipping.
- B. Results: At the request of W. G. Sanderson, peel tests were performed with the Dynastat to rank three adhesives used in overtipping. Nine adhesive-tipping paper combinations were tested. In none of these cases was there bond failure due to the breakage of the glue. Failure in all cases was due either to paper delamination at the adhesive interface or tensile failure of the paper itself. A memo summarizing the results was issued.
- C. Plans: No further work is planned.

IV. MENTHOL STUDIES (D. Driscoll)

- A. Objective: Determine the diffusion coefficient of menthol in cellulose acetate (CA).
- B. Results: Experiments to determine the permeability of CA films with respect to menthol have been initiated. The permeability of

unplasticized CA films at room temperature was found to be negligible while a slight, but measurable, permeation rate was observed at 40°C. The results to date indicate minimal mobility for menthol in unplasticized CA.

C Plans:: This work is ongoing.

PROJECT NUMBER: 1720
 PROJECT TITLE: Analytical Microscopy
 PROJECT LEADER: V. L. Baliga
 PERIOD COVERED: October, 1988

I. PROJECT ART (Baliga, Sanders, Henry)

A. Objective: Characterize changes to the epidermal surfaces of super-critically extracted (SCE) tobaccos and determine degree of shrinkage.

B. Results:

Super-critically extracted tobacco samples taken from the top of the basket were covered with a heterogeneous waxy material that melted between 34°C to 60°C. The waxes were demonstrated with the use of a lipid stain, Sudan IV. The white crystalline material on the tobacco shreds melted at about 60°C, similarly to that of pure solanesol. The yellowish greenish material which also coated the shreds, melted at around 34°C, similarly to that of a crude preparation of solanesol prepared from materials from the DIET expansion process.

The cuticle layers of tobacco shreds taken from the top of the basket, the bottom of the basket, and the unextracted control were examined for structural differences. All samples contained bumps and ridges as well as small hemispherically raised regions that could be 'blisters' or wax deposits. One difference was noted in the unextracted control. The epidermal cells of the control did not appear as collapsed along the plane parallel to the surface of the leaf as did the SCE samples. However, when cross sectional thicknesses were measured to compare sample shrinkage, there were no differences among the SCE samples and the controls as noted in the table.

<u>Sample</u>	<u>thickness μm (SD)</u>		<u># shreds measured</u>
Unextracted control blend	72	(19)	15 ¹
SCE, top of basket blend	95	(27)	14 ¹
SCE, bottom of basket blend	79	(24)	20 ¹
Unextracted thin burley	59	(19)	5 ²
SCE thin burley	49	(18)	5 ²

There also was no surface shrinkage of the epidermal cells. This was measured by counting stomata per unit area from bodied burley before and after SCE³.

<u>Leaf Surface...top (SD)</u>	<u>Unextracted, bodied burley</u>		<u>SCE, bodied burley</u>	
	<u>bottom (SD)</u>		<u>top (SD)</u>	<u>bottom (SD)</u>
# of stomata per 0.25mm ² ..	13 (12)	44 (18)	12 (9)	35 (14)

C. References:

1. Baliga, V., and Henry, B., "Characterization of Super-Critically Extracted Tobaccos," memo to Sue Wrenn, October 14, 1988.
2. Sanders, K., P.M. Notebook # 8612, p. 189.
3. Sanders, K., "Structural Examination of ART Tobaccos," memo to V. Baliga, October 13, 1988.

II. SUPPORT TO OPERATIONS AND R&D (Sanders)

A. Objective: Provide analytical support to Operations and R&D.

B. Results: In an effort to recognize corrosion on metal surfaces in the ART extractor, three C-rings were examined and photographed to document their appearance before they were installed in the ART extractor. The surfaces of all samples contained striations made during the machining process. Also, there appeared to be a coating layer of metal that had partially separated from its underlying layer. Differences that were unique to individual samples included a smooth surface on one and a granular surface on another. None, however, were pitted from corrosion¹.

C. References:

Sanders, K., "Analytical Microscopy Result Form," to B. Laroy, September 21, 1988.

III. LOW SIDE STREAM (Baliga, Henry)

A. Objective: Examine the structure of cigarette papers and paper additives.

B. Results: A memo was issued that documented the characterization of five handsheets made by S. Baldwin¹. This had been summarized in the September monthly.

C. Reference:

Baliga, V., and Henry, B., "Structural Characterization of CaCO₃ Handsheets", memo to S. Baldwin, October 13, 1988.

IV. MICROSCOPY TECHNIQUE (Henry)

A. Objective: To Improve photographic procedures for the AMRAY scanning electron microscope (SEM)

B. Results: An improved photographic procedure was developed for samples that have a tendency to build up an electrical charge during the SEM photographic process. A more rapid scan rate was used during the film exposure which shortened the interaction time of the electron beam with the sample¹.

C. References:

Henry, B., "Use of Scan Rate to Enhance Detail of Photomicrographs," memo to V. Baliga, October 5, 1988.

V. MISCELLANEOUS (Sanders, Baliga)

A. Results:

K. Sanders attended a class on the use of polarized light microscopy in Chicago, Illinois, from September 12 to 16, 1988.

V. Baliga attended TCRC at Lexington, Kentucky, October 3 to 5, 1988.

VI. SAFETY (Sanders)

A. Objective: Provide instruction to ERT members on various aspects of first aid.

B. Results: One ERT class was given on the recognition and treatment of shock.

PROJECT NUMBER: 1730
PROJECT TITLE: Plant, Cell & Tissue Culture Research
PROJECT LEADER: I. L. Uydess
WRITTEN BY: L. Weissbecker
PERIOD COVERED: October, 1988

I. TOBACCO-IDENTICAL PRESERVATIVES

A. Objective: To develop procedures and to establish microbiological screens for the evaluation of new, nature-identical preservatives as replacements for and/or as adjuncts to propylparaben.

B. Status:

Phase I Screens:

Potassium sorbate has been evaluated in the Phase I shake-flask assay against propylparaben, benzoic acid and decanoic acid for antimicrobial activity against *B. coagulans* (PM-13). Potassium sorbate at 1000 µg/ml produced a half-maximal inhibition of growth similar in effect to that of the 150 µg/ml paraben control and 500 µg/ml benzoic acid. The 50 µg/ml decanoic acid control completely inhibited the growth of the target organism, PM-13.

An assay to assess the efficacies of a decanoic acid-propylparaben mixture and of a decanol-propylparaben mixture (each component at a 25 µg/ml level) was performed. The decanol-propylparaben mixture completely inhibited bacterial growth of PM-13 while the decanoic acid-propylparaben mixture was not completely inhibitory. The greater solubility of decanol probably accounts for these results.

Phase II Screens:

A dose-response assay applying decanoic acid in Park 500 SEL was conducted in the Phase II screen. It now appears evident that 300 to 400 µg/ml of decanoic acid provides approximately the same antimicrobial activity with regard to inhibiting many of the chemical (including pH) changes that accompany spoilage as the 600 µg/ml level used in previous experiments.

Phase III Screens:

SEL fermentor experiments with decanoic acid were run at 37°C and 50 rpm using Park 500 SEL. Initial results indicate that decanoic acid exhibits the same antimicrobial effect in 5-liter fermentors as it does in 100 ml shake flask experiments. No change in pH, total reducing sugars, formic acid, acetic acid, NO₃⁻ and NH₃ were observed over a 24-48 hour period in the fermentor containing 600 µg/ml decanoic acid. Significant changes were observed for all the variables in the control fermentor containing the fresh, Park-500 SEL.

Miscellaneous:

A pilot plant run with decanoic acid has been completed (week of Oct. 10th). TC and 150B sheets +/- decanoic acid at final concentrations (on the sheet) of 1000, 2000 and 4000 ppm decanoic acid were produced for subjective evaluation.

C. Conclusions:

1. Potassium sorbate and benzoic acid were not as effective in the Phase I screen against PM-13 as were propylparaben or decanoic acid. Decanoic acid, at 50 µg/ml was found to be the most effective antimicrobial agent tested.
2. Three hundred to four hundred ppm decanoic acid in Park 500 SEL appears to be as effective in inhibiting spoilage in the Phase II screen as was the 600 ppm level decanoic acid previously reported.

D. Plans: October, 1988

1. Further evaluations of the fatty acid alcohols in conjunction with fatty acids will be conducted.
2. The C-10 and C-12 fatty acid alcohols will be tested in the Phase II, SEL shake-flask assay.
3. Evaluations of the C-10 and C-12 fatty acids and fatty acid alcohols will be initiated in the Phase III fermentor screen.
4. Dissolved oxygen, pH and temperature measurements will be made by Project 1730 and Park 500 personnel at selected locations in Line 2 of Park 500 in support of the design and establishment of a Park 500 model system at the R&D labs.

PROJECT NUMBER: 1752
PROJECT TITLE: Optical Spectroscopy of Tobacco and Smoke
PROJECT LEADER: J. O. Lephardt
PERIOD COVERED: October, 1988

I. MASS SPECTRAL ANALYSIS OF WAXES

- A. Objective: To develop mass spectral methods utilizing soft ionization and MS-MS techniques for analysis of waxy materials and mixtures of waxes.
- B. Results: Sample preparation is in progress. In addition to obtaining samples of waxy material from tobacco, several model systems are being prepared. These model systems will be used as markers to identify the most expedient analytical approaches, to facilitate interpretation of data obtained from natural mixtures, and in some cases to provide analytical selectivity towards compounds of particular interest.
- C. Plans: Samples will be taken to a tandem mass spectrometry facility for analysis in mid November.

II. THERMAL ANALYSIS

- A. Objective: To establish capability in the Analytical Division for performing Thermal Gravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) measurements.
- B. Results: A Perkin-Elmer TGA-7 and a Perkin-Elmer DSC-7 instruments have been installed in B-216 and Charles (Chuck) Chung has been hired to perform analyses with these instruments. Training by Perkin-Elmer of Chuck and Jim Lyons-Hart on the care and use of these instruments will occur during the first week of November.
- C. Plans: The initiation of sample analyses using these instruments is intended to occur during the second week of November.

III. MULTICOMPONENT SMOKE ANALYSIS (MSA) OF ART CIGARETTE MODEL

- A. Objective: To evaluate MSA data for differences between control and sample.
- B. Results: For both models, two sets of three cigarettes each were smoked on a standard five port cigarette machine. The Nicolet 160sx FT-IR was used as the detection instrument. The peak areas of 14 gas components were calculated. These data were sent to the VAX system and placed in a RS1 format. RS1 tables and graphs were created for data analysis. Both sets of control and sample data were averaged and compared by statistical programs in RS1.

- C. Conclusions: A review of the component areas reveals that no significant differences are apparent for any of the gases.
- D. Plans: A memo will be written to Betty Handy detailing the above work and results.

PROJECT NUMBER: 1754
PROJECT TITLE: Spectroscopic Studies of Tobacco and Smoke Components
PROJECT LEADER: J. B. Wooten
WRITTEN BY: R. L. Bassfield
PERIOD COVERED: October, 1988

SOLUTION NMR

- A. **Objective:** Use NMR to determine the optical purity of extracted nicotine.
- B. **Results:** The first of two samples submitted by R. Izac for optical purity analysis contained impurities which caused spectral broadening upon the addition of the chiral shift reagent. Therefore a purity measurement was not obtainable. The second sample, free of impurities, displayed good resolution over the course of the additions of shift reagent and no splitting of the N-methyl resonance was observed.
- C. **Conclusions:** This would indicate that the second sample was one isomer or predominantly one isomer; since a small amount of one might be hidden in the base of the taller peak.
- D. **Plans:** A novel approach to optical purity measurements of alkaloids, by NMR, was recently reported. This procedure is based on the inclusion of these compounds with β -cyclodextrin which causes differences in the chemical shifts of the two enantiomers. It is reported that little or no broadening occurs with resolution at several sites. If successful, we plan to use this procedure as a model for our nicotine determinations.
- A. **Objective:** Determine the structure of an unknown glucoside isolated from bright tobacco.
- B. **Results:** ^1H , ^{13}C and 2D NMR experiments have been employed to identify this compound as a sesquiterpene glucoside with the aglycone possibly derived from solavetivone. Comparison of our data with a literature reference was favorable. Preliminary 2D results indicate the site of the glucoside linkage is the carbon atom adjacent to the carbonyl in the cyclohexenone ring, and not on the sidechain as expected.
- C. **Plans:** Additional experiments will be performed in order to confirm the site of the glucoside linkage.

PROJECT NUMBER: 1757
PROJECT TITLE: Analytical Flavor Specifications
PROJECT LEADER: M. L. Zimmermann
PERIOD COVERED: October, 1988

ANALYTICAL FLAVOR SPECIFICATIONS

- A. Objective: To develop analytical and sensory specifications for incoming flavors and materials for use at the Flavor Center and other QA facilities.
- B. Results: Work continues towards the completion of the second vendor specifications. Nearly three-quarters of the materials received have analytical and subjective evaluations completed. Receipt of the remainder of the samples is anticipated.

Scheduled PMI shipment lot samples have been requested to initiate certification of materials for compliance to German standards. Some of the reference materials are still being located.

A sample of artificial wine flavor was analyzed by GC, IC, GC/MS, and GC/FTIR to determine its composition.

A paper was presented at the 9th International Tobacco Scientific Congress in Guangzhou, China.

- C. Plans: Complete specifications for the second vendor and continue with the work on the PMI samples.

PROJECT NUMBER: 1758
PROJECT TITLE: Tobacco Cell Wall Research
PROJECT LEADER: G. H. Bokelman
WRITTEN BY: Sheryl Baldwin
PERIOD COVERED: October, 1988

I. SIDESTREAM REDUCTION (S. Baldwin, B. Goodman, J. Paine and B. Rogers)

A. Objectives: (1) To prepare handsheets using inorganic paper fillers with different physical and chemical properties to be evaluated for effectiveness in sidestream smoke reduction; (2) to prepare coated bobbins of paper for the same purpose; (3) to obtain new or different paper-making materials of possible value in sidestream smoke reduction.

B. Results: A series of 24 handsheets were prepared at the University of Maine with calcium carbonate fillers covering a range of particle sizes from 0.02 microns to 2 microns, with surface areas of 7 m²/g to 72 m²/g. Physical properties of the sheets have been determined. Permeability matches were obtained for three sets of handsheets with fillers of very different surface areas. Additionally, handsheets with the same Coresta permeability were prepared with both calcium carbonate and magnesium carbonate of the same particle size. Preliminary sidestream reduction results with magnesium carbonate had shown promise.

Samples of alternate counterion CMC have been received from Aqualon. Potassium, calcium, magnesium and ammonium ions have been substituted for sodium in carboxymethylcellulose. Two bobbins of paper obtained from Kimberly Clarke were treated with potassium succinate and coated with two types of carboxymethylcellulose (CMC). One bobbin was coated with standard sodium CMC as a control. The other bobbin was coated with a new product in which the NH₄⁺ ion completely replaces the sodium.

A number of cellulose derivatives were suggested as possible paper coatings to provide spotting protection for cigarettes prepared from ART tobacco.¹

A laboratory size coating unit has been ordered to apply sizing and other materials to the handsheets. Rolls of paper produced at the University of Maine were split and prepared for splicing into bobbins suitable for coating and running on the maker.

Refining curves were repeated in the new handsheet facility. Flax fiber was regenerated from cigarette paper, but the porosity of the resulting sheet was higher than that of the parent sheet. Fines may have been lost, or the pH may not have been high enough to adequately swell the fibers.

A sample of a precipitated dolomite with a high surface area, 26 m²/g, has been received from Pfizer. A sample of a slurried high surface area carbonate has also been received. Samples of aluminum hydroxide magnesium carbonate, hydrotalcite, and

magaldrate have been submitted for SEM examination to determine particle size and structure.

A database has been established to retain the data for the various papers prepared to date.

C. **Plans:** The handsheets prepared will be chemically treated, pending the arrival of the coater. Cigarettes will then be prepared for evaluation for light extinction. Eight additional bobbins of paper will be prepared at the University of Maine for coating with the alternate counterion CMC's and with the thermoplastic cellulose derivatives. Hand sheets will also be prepared with the new inorganic fillers which have been received.

D. **Reference:**

1. Baldwin, S., memo to W.G. Houck, "Suggestions to Address the Spotting Problem in ART Cigarettes," October 7, 1988.

II. **ESTIMATION OF BLEND COMPOSITION FOR CARTIER AND FRONTIER CIGARETTES**
(G. Bokelman, J. Stimler and General Analytical)

- A. **Objectives:** As requested by W.G. Houck, determine the estimated blend composition of two brands of Cartier, Regular and Pearl Tipped. Also, as requested by C.R. Lambert, predict the blend composition of three new brands of cigarettes introduced on the Japanese market by Japan Tobacco, Inc. (JTI): Frontier Regular, Frontier Menthol, and Frontier Lights.
- B. **Results:** Cartier cigarettes were of interest because they have an extremely low incidence of spotting. The predicted blend compositions for the Cartier cigarettes are shown in Table 1. These two brands are notable for their high contents of bright lamina and absence of Oriental leaf. It appears unlikely that the "secret" of the lack of spotting by Cartier cigarettes is somehow related to a novel or unusual blend composition. However, these tobacco blend components could be processed in a different manner.

Table 1. Predicted Blend Compositions for Cartier Cigarettes
(values expressed as percentage of total)

<u>Component</u>	Cartier	Cartier
	<u>Regular</u>	<u>Pearl Tipped</u>
Bright Lamina	62	68
Burley Lamina	15	17
Oriental Leaf	0	0
Stem	23	15

The predicted blend compositions for the three new cigarette brands from JTI are shown in Table 2. It is apparent that all three brands have the same tobacco blend composition. This one common blend is similar to that of SomeTime Lights (from JTI) and Marlboro Lights (Japan). Compared to SomeTime Lights, the Frontier blend contains more burley lamina and Oriental leaf, but it has a lower content of (combined bright and burley) stems.

Table 2. Predicted Blend Compositions for New Cigarettes Introduced by
JTI
(values expressed as percentage of total)

<u>Component</u>	<u>SomeTime</u>	<u>Frontier</u>	<u>Frontier</u>	<u>Frontier</u>
	<u>Lights</u>	<u>Regular</u>	<u>Menthol</u>	<u>Lights</u>
Bright Lamina	41	42	43	40
Burley Lamina	29	32	32	33
Oriental Leaf	6	8	10	8
Stem	25	18	16	18

- C. Plans: No additional cigarettes are currently being evaluated for blend composition estimation.

III. CHARACTERIZATION OF TOBACCO HEMICELLULOSE (S. Baldwin)

- A. Objective: Characterize hemicellulose isolated from green Coker 319 tobacco.
- B. Results: As part of a collaborative contract research project, Dr. Norman Lewis at VPI&SU sent us a crude sample of hemicellulose that had been extracted from green Coker 319 tobacco grown in the greenhouse at Philip Morris. Recently an additional three samples of tobacco hemicellulose were received. Based on differences among the gel permeation chromatograms of these samples and differences in their neutral sugar analyses, it was concluded that these samples consist of differing ratios of the same, or in some cases slightly different, components. Fractions believed to represent different components were collected. Neutral sugar analyses confirmed that these were indeed different components. The components were found to have the following compositions:

1. arabinose:xylose:galactose:glucose molar ratios of
1: 7.8:1.35:21.2
2. rhamnose:arabinose:xylose:galactose:glucose molar ratios of
1:1.67:3.2:3.2:10.2

Methylation analysis of the original unseparated hemicellulose sample and on the more soluble components revealed the following:

Original sample: t-arabinose, t-xylose, t-glucose, t-galactose, 1,4-glucose, 1,4-galactose, 1,4-xylose, 1,4,6-mannose, and 1,4,6-glucose.

"Soluble sample": t-arabinose, t-xylose, t-glucose, t-galactose, 1,4-glucose, 1,4-galactose, 1,4-xylose,

- C. Plans: The individual components within these various samples of tobacco hemicellulose will be isolated and more completely characterized.

IV. EVALUATION OF FLAVOR BEADS FROM PREMIER (S. Tafur)

- A. **Objectives:** Determine whether deoxyfructosazines (DOF's) were present in Premier flavor beads.
- B. **Results:** Nicotine was extracted from the flavor beads from 40 Premier cigarettes in order to check its enantiomeric purity.¹

Water and methanol extracts of the flavor beads of Premier cigarettes were prepared and examined by LC for detectable levels of deoxyfructosazines (DOF's). These compounds are considered indicative of the presence of some reaction flavors involving fructose and glucose. No definitive peaks were observed in the expected retention region for DOF's. Further examination of the extracts for tobacco glycosides which might occur in a tobacco extract used as a flavorant did not indicate any appreciable level of glucosides.²

- C. **Plans:** No further work is planned.

D. **References:**

1. Core, M., Izac, R., Tafur, S., "Enantiomeric Purity of Nicotine from Premier Flavor Beads," memo to R. Ferguson, October 10, 1988.
2. Tafur, S., "LC Examination of Extracts of Premier Flavor Beads," memo to R. N. Ferguson, October 14, 1988.

PROJECT NUMBER: 1902
PROJECT TITLE: Tobacco Microbiology
PROJECT LEADER: D. J. Ayers
WRITTEN BY: O. M. Gaines
PERIOD COVERED: October, 1988

I. SHREDDED STEM STUDY (FOR PROJECT ART)

- A. Objective: To determine if pre-ART bright shredded stems with citrate spray (at 35 %OV) and post-ART "as is" stems (at 35 %OV) show a change in microbial numbers after 24 hours of storage at 35°C and to determine if post-ART dried stems (at 12 %OV) show a change in microbial numbers after 14 weeks of storage at 35°C.
- B. Results: Bacterial counts did not increase, over initial counts, in the pre-ART stems with citrate and the post-ART stems at 24 hours of storage. A ~ 1 log increase, over initial counts, was seen at week 1 in post-ART dried stems but counts decreased below initial counts at 14 weeks of storage. Mold and yeast were within acceptable limits for the pre-ART and post-ART stems at week 14. Mold counts remained within acceptable limits but yeast counts exceeded acceptable limits at weeks 0 and 14 in post-ART dried stems.
- C. Plans: Issue a memo reporting the results.
- D. Reference:
1. Jones, J. Notebook No. 8590, pp. 59-61, 73.

II. YEAST ENCAPSULATED PEPPERMINT OIL (YEPO) STUDY

- A. Objective: To test a sample of yeast encapsulated peppermint oil for viability and bacterial contaminants.
- B. Results: There were no viable bacterial, mold or yeast cells/gram detected in the sample.
- C. Plans: Issue a memo reporting the results.
- D. Reference:
1. Gaines, O. Notebook 8690, pp. 53, 69-74.

III. BACTOMETER CALIBRATION CURVES

- A. Objective: To generate calibration curves for determining the number of bacteria present in a sample using the Bactometer.
- B. Results: Statistical analyses indicated that RL-TC, RL-150B, and Burley can be run on the same calibration curve. Bright, Oriental, and Blend tobaccos require separate curves.

C. Plans: Complete data collection for the completion of calibration curves for Bright, Oriental, and Blend.

D. Reference:

1. Chadick, D. Notebook 8625, pp. 158-161.

IV. DRY-PUFF CIGARETTE STUDY

A. Objective: To determine if the transmission of bacteria occurs after a cigarette is dry-puffed.

B. Status: Experiments were conducted using an aseptically designed apparatus. 1R4F cigarettes were dry-puffed, and the air passed through the cigarette was collected in sterile saline. The saline, dry-puffed and non-dry-puffed cigarettes were analyzed for bacterial growth.

C. Results: No bacterial growth was detected in the saline. The bacterial counts obtained from the dry-puffed cigarettes were similar to the counts from non-dry-puffed cigarettes.

D. Plans: No further experiments are planned unless requested.

E. Reference:

1. Chadick, D. Notebook 8625, pp. 150-153, 162.

V. ART FILLER STORAGE STUDY

A. Objective: To determine the effects of storage on the microbial load of ART filler at 35°C with 80% RH.

B. Results: Bacterial counts showed no increases over initial counts for the data accumulated from experiments 1-5 after 2-8 weeks of storage. The mold and yeast counts were variable and the majority of them exceeded acceptable laboratory limits (≤ 80 mold and/or yeast colonies per gram, see Reference 1) in all five experiments to date (2).

C. Plans: Continue study.

D. References:

1. Crockett, E. A. The Microbial Quality Improvement Program (MQIP) As Conducted in the OC Semi-Works Primary Facility. Special Report #87-155; 1987 December 21.
2. Jones, J. Notebook 8590, pp. 73, 75, 77-80, 83.

VI. CTB AND BURLEY SPRAYED BURLEY STORAGE STUDY

- A. Objective: To determine if the addition of class tobacco dust (CT) to RBS would enhance microbial development on the Burley strip after 12 weeks of storage at 35 C with 80% RH.
- B. Results: The storage study is complete through 4 weeks. The bacterial, mold and yeast counts are within the laboratory limits.
- C. Plans: Continue study through 12 weeks of storage.
- D. Reference:
 - 1. Chadick, D. Notebook 8625, pp. 133-138, 141-142, 149, 154.

PROJECT NUMBER: 1904
PROJECT TITLE: Tobacco Physiology and Biochemistry
PROJECT LEADER: H. Y. Nakatani
WRITTEN BY: H. Y. Nakatani and E. D. Mooz
PERIOD COVERED: October, 1988

I. LOW NICOTINE STUDY

- A. **Objective:** To investigate the biochemistry of the nicotine biosynthetic pathway at putrescine N-methyltransferase (PMT) and specifically to isolate PMT from tobacco root extracts.
- B. **Status:** PM extracts from the ammonium sulfate stage were processed through the two column series of phenyl-Sepharose and DEAE-Sepharose columns. The specific activity of the pooled fractions after the DEAE-Sepharose stage was > 600 units for one preparation. The pooled fractions from the DEAE-Sepharose column following a second series was separated into two parts in which the specific activities were found to be > 300 and > 600 units (1,3,6). A unit is equivalent to a nmole of S-adenosylmethionine converted per mg protein in 30 minutes at 30°C.

A number of affinity support media were further tested for possible use in the purification of PMT; these included ω -aminohexyl Sepharose 4B (AHS), ω -aminopropyl Agarose (APA) and the ω -aminoethyl Agarose (AEA) materials (5,6). Thirty ml of a PMT sample (0.2 mg/ml) was applied to the AHS column in an attempt to concentrate the PMT; however, the elution volume upon removal of PMT with 1 M NaCl was 10-12 ml, indicating that this method is not appropriate to concentrate PMT. Only 15% of the PMT applied to the APA column was eluted upon addition of a 0-30 mM putrescine gradient. More emphasis was therefore concentrated on the AEA column. PMT activity appeared to be separated from the majority of the protein near the beginning of the salt gradient (0-50 mM and 0-100 mM NaCl) with this medium. Moreover, either washing with 10 mM NaCl or 5 mM putrescine was found to be sufficient to elute bound PMT from this support medium. Six mg of protein was determined to be the maximum loading of a 3 ml bed volume column (5,6). Several protein bands, however, were still observed after examination of the PMT sample eluted with putrescine by SDS-PAGE (3,5,6). Work is continuing to find conditions ("affinity") to elute PMT more specifically.

A number of other affinity gels (prepared by Dr. W. P. Hempfling) coupled to N-methylputrescine (NMP) were also examined. PMT was bound to the NMP-aminoethyl-1,6-diaminohexyl Agarose and NMP-CH Sepharose 4B columns. However, no PMT activity was eluted with gradients of the substrates, SAM (0-5 mM) or putrescine (0-30 mM). PMT activity was eluted in an apparent ion exchange mode upon additional application of a 0-500 mM NaCl gradient. The PMT activity eluted with the majority of the protein in the 250-300 mM NaCl concentration range (4). Inconclusive results were obtained from a column in which NMP was coupled to the aminoethyl Agarose support medium (4).

Further examinations of S-adenosyl-homocysteine (SAH) linked to the 1,6-diaminohexane Sepharose support medium were also conducted (prepared by Dr. W. P. Hempfling) (2). Initial examination showed that some PMT could be eluted upon application of a putrescine gradient (0-50 mM); however, the elution of PMT appeared to be non-specific (possible ion-exchange mode) (2,3). More recently, some PMT was found to be eluted with 1 mM SAM and the remainder upon application of 500 mM NaCl. The SDS-PAGE profile of the PMT sample eluted with 1 mM SAM showed a relatively clean profile with a major band having an apparent molecular weight between 55 and 65 kDa whereas the PMT sample eluted with salt was highly contaminated with a number of protein bands (2,3). These results suggest that sites exist on the affinity material which can bind to PMT in an ion-exchange as well as affinity mode. New gels are being prepared with the use of ethanolamine to reduce the number of ion-exchange sites.

Native gel (ELFE) examination of PMT was also continued. The gel with 10% acrylamide and 10% glycerol appears to give the best results; elution time was reduced considerably with 12% acrylamide (3). Various concentration methods (Amicon, Centriprep and Lyphogel) were also examined. The Amicon and Centriprep methods appear to be the most useful at this time (3).

- C. Plans: To block ion-exchange sites on affinity matrices for purification of PMT. To focus on the elution of PMT from the affinity matrices, by eluting PMT with substrate conditions. To examine possible increase in the resolution of the native gel (Canalco) system to purify PMT by increasing the length of the gel. Continue ³H-SAM labeling studies. Obtain PMT-active samples for application to affinity and native gel.

D. References:

1. Dunn, R. L. Notebook No. 7899.
2. Malik, V. Notebook No. 8542.
3. Davies, S. Notebook No. 8694.
4. Yu, T. Notebook No. 8381.
5. Mooz, E. D. Notebook No. 8599.
6. Crockett, E. Notebook No. 8563.

II. ALTERNATE HUMECTANTS (PG/G-FREE SHEETS/CIGARETTES) (1)

- A. Objective: To produce an acceptable full-flavored cigarette which is PG/G-free by the end of 1988 and to send it and a suitable control cigarette out as a POL test.
- B. Results: A request for another PG/G-free cigarette model has been made to the Flavor Development Division personnel (2).
- C. Plans: The second PG/G-free cigarette model will be evaluated analytically and subjectively. Control feedstocks and PG/G-free

RL, RCB, ESB, and DIET will be re-evaluated analytically, microbiologically, and subjectively prior to making PG/G-free POL cigarettes.

D. References:

1. Mooz, E. D. Notebook No. 8599.
2. Ruziak, S. Personal Communication to E. Mooz. 1988 October 19.

III. ALTERNATE HUMECTANTS (GLYCERINE-FREE SHEET/CIGARETTES) (1)

- A. Objective: To produce an acceptable glycerine-free (G-free) cigarette (domestic Marlboro-type) for a POL test by the end of the first quarter of 1989.
- B. Results: The G-free program has been put on hold pending the outcome of the PG/G-free effort.
- C. Plans: There are no further plans at this time.
- D. References:
1. Mooz, E. D. Notebook No. 8599.

PROJECT NUMBER:: 2106
PROJECT TITLE:: Cigarette Performance and Design
PROJECT LEADER:: R. W. Dwyer
PERIOD COVERED:: October, 1988

I. FILTER RESEARCH AND DEVELOPMENT

- A. Objective: Design novel filters for achieving subjective advantages with new products.
- B. Results: A concentric filter design was developed for the Art Program. The filter was designed to provide primarily peripheral flow at the 11-mg tar delivery level. A computer-based concentric filter design program was written and provided to the Filter Research and Development Project. These routines allow the user to select tow items for the inner and outer filter segments based on the desired flow profile, ventilation level, and filter efficiency.
- C. Conclusions: The limited delivery data available for concentric filters suggest the current computer programs are adequte for predicting flow paths and tar deliveries. New designs have been submitted to Filtrona in order to collect additional data to validate the model.
- D. Plans: A patent disclosure was submitted to our patent group concerning a type of concentric filter design. The design involves balanced resistances to flow in both sections, but with the sections having different efficiency materials. In this manner the delivery of core or peripheral smoke from the tobacco rod could be selectively delivered.
- E. References: This work is being done in conjunction with the Filter Research and Development Project.

PROJECT NUMBER: 2500
PROJECT TITLE: Fundamental Chemistry
PROJECT LEADER: J. I. Seeman
PERIOD COVERED: October 1988

I. FLAVOR/ODOR CHEMISTRY (Houminer, Paine, Secor, Seeman)

- A. Objective: To develop new technologies for smoke deliveries of desired flavorants; to prepare new substances for flavor/odor evaluation.
- B. Results and Plans: A major effort has begun regarding the optimization of the preparative methodologies for polyMIC, poly[isopropenylmenthylcarbonate], a menthol release agent. A literature search on alternative preparations and alternative release agents has been completed. The reaction of acetone + KO-tert-butoxide + menthylchloroformate under various conditions gave the desired menthyl isopropenylcarbonate in addition to menthyl tert-butylcarbonate in a ratio of ca. 4:1. Experimental conditions are being varied to improve the yields and product ratio. The reaction of diacetone alcohol with methyl Grignard did not yield the desired precursor to a polyMIC analogue.

Nicotine extracted from a Premier was examined and shown to be the natural (S)-(-)-isomer. A previously examined sample was found to be contaminated with an artifact.

II. CHEMICAL PHYSICS STUDIES OF TOBACCO CONSTITUENTS (Seeman)

- A. Objective: To obtain structural information about important tobacco constituents/flavorants; to develop information on cluster formation and chemical reactions in clusters.
- B. Results and Plans: Three analogues of the tobacco alkaloids (benzylamine, N-methylbenzylamine, and N,N-dimethylbenzylamine) are being examined under laser jet spectroscopic conditions, both as free molecules and in clusters with CO₂ and CH₄. Dual beam experiments are being considered for chemical reactions in clusters.

III. REMOVAL OF NICOTINE FROM AQUEOUS TOBACCO PROCESSING FLUIDS (Hassam, Seeman)

- A. Objective: To develop techniques to remove nicotine and other tobacco alkaloids from aqueous tobacco processing fluids to the exclusion of all other components.
- B. Results and Plans: Various items have been purchased and space has been readied for the imminent arrival of the Sepracor membrane system early next month. To establish a model system for use in the extractions, solubilities and gc analyses have been determined for a variety of components found in the aqueous scrubber liquid.

IV. MISCELLANEOUS (Houminer, Hassam, Secor)

- A. Results: A pentylated β -cyclodextrin was prepared for use in the GC analysis of tobacco enantiomers. Various TSNA-- ^{14}C -NNK investigations have been completed, mostly dealing with analytical methodology development. The first kinetic run of the thermolysis of a number of pyrazineethanols has been completed and a duplicate run is in progress and near completion.

PROJECT NUMBER: 2501
PROJECT TITLE: Smoke Chemistry
PROJECT LEADER: R. Comes
PERIOD COVERED: October 1988

I. SIDESTREAM SMOKE

A. Objective: Conduct studies on sidestream smoke including: development of methods for collection and analysis of sidestream gas phase and semivolatiles; visibility determinations; analysis of selected materials relating to sidestream odor and irritation; development of potential proprietary products.

B. Status: (1) Methods development for analysis of sidestream smoke components continues. (2) Construction of instrumentation to monitor visibility of sidestream smoke has been ongoing. Visibility measurements are run as required. (3) Studies have proceeded in the design and optimization of an apparatus for CORESTA sidestream measurements. Selected samples have been run in support of this effort.

C. Results: (1) The Tekmar unit used for analysis of sidestream smoke has been disconnected from the gc/ms. Direct injection of concentrated solvent samples of sidestream smoke has been investigated while building and developing a valve-controlled heart-cutting apparatus. Compounds can now be transferred from the primary to the secondary column. Operational problems still need to be addressed. A sidestream smoking chamber with a trap that can accommodate smoke from thirty cigarettes has been fabricated for investigation of components present in very low concentration. (2) The 8-port visibility apparatus has been placed in operation and a series of Monitor 25 cigarettes run as a system test and to establish operating procedures. Problems relating to the "pooling" and "curling" of smoke at the outer edges of the hood have been addressed. (3) Results of CI data from 1R4F cigarettes run on the "Enclosed Fishtail" CORESTA smoking machine at sidestream flow rates of 2, 3 and 4 liters/min have been obtained and reported. An additional sample in the low density rod program has been obtained and smoked on the CORESTA apparatus. Results support the conclusions discussed in the Sept. 88 Monthly Report. Some core/periphery studies have been initiated to determine the effect of replacement (modification) of these areas in a cigarette on the mainstream/sidestream deliveries. Surprisingly, an increase in nicotine delivery to both mainstream and sidestream is observed in the cigarettes with a center core of "pith" as compared to the delivery for a normal 2R1 cigarette.

D. Plans: (1) The total Tekmar system will be set-up after optimization of the heart-cutting procedure is completed. The analysis of the compounds present in very low concentration in sidestream smoke will commence shortly. (2) Additional modifications to the 8-port visibility apparatus will be made and smoking runs for system check-out will continue. (3) Core/periphery studies will proceed utilizing other materials to attempt to modify mainstream/sidestream delivery ratios.

II. MISCELLANEOUS

A. Nicotine analyses have been carried out in support of Analytical Research and the ART project. A comparison of the internal standards n-butylornicotine and isoquinoline (standard used in method #E-86) in gc nicotine analyses gave excellent agreement. Efforts will continue for improvement of accuracy and precision of the analysis, determination of factors affecting quantitation in actual samples and extension of the procedure to other alkaloids.

B. Samples of pre-ART filler and ART processed filler treated by vacuum drying are being investigated by gc to determine if differences are detectable. This work is in support of the attempts to alleviate the "spotting" problems with ART filler.

C. Investigations of the RJR Premier cigarette have been carried out. An initial study utilizing ^{14}C -nicotine applied to the flavor beads and separately to the tobacco filler showed a 6% transfer from the beads and only a 0.2% transfer to mainstream from the filler. Recovery of applied ^{14}C -nicotine was only 50-55%. These investigations are continuing.

GC and GC/MS evaluation of both the flavor beads and smoke have been initiated. Some bead flavor components have been identified and many smoke constituents determined. Additional identifications are ongoing as different collection and elution techniques are utilized.

D. Several GC and GC/MS analyses were conducted in support of Division needs. The new Finnigan SSQ-70 mass spectrometer has been received, installed and is currently being tested using various ionization techniques.

PROJECT NUMBER: 2520
PROJECT TITLE: Flavor Research
PROJECT LEADER: E. W. Southwick
PERIOD COVERED: October, 1988

I. GLUCOSE-DERIVED FLAVORANTS

- A. Objective: To develop glucose-derived flavorants.
- B. Results: A sample of menthol encapsulated in sodium alginate was obtained by spray-drying a dilute aqueous solution of sodium alginate and menthol. The amount of encapsulated menthol was relatively low. Efforts to increase the loading are being pursued.

II. PROJECT EXTRA

- A. Objective: To develop proprietary flavor additives for enhanced flavor perception in low delivery cigarettes.
- B. Results:
- (1) An additional large scale (10g) synthesis of 6-methyl-hexahydro-2(3H)-benzofuranone (CR-2643) has been completed to support Flavor Development. As a result of this work, the three-step procedure has been improved to give an overall yield of greater than 70% in as little as two working days.
 - (2) Catalytic reduction of ethyl 2-cyclohexanoneacetate followed by cyclization gave 1.0 gram of hexahydro-2(3H)-benzofuranone. This will be evaluated subjectively when time permits.
 - (3) The synthesis of a series of hydroxycyclopentenones at VCU has been completed. As a result, we have on hand the following smoke components:

3-ethyl-2-hydroxy-2-cyclopenten-1-one
3-(1-propyl)-2-hydroxy-2-cyclopenten-1-one
3-(1-butyl)-2-hydroxy-2-cyclopenten-1-one
3-(2-butyl)-2-hydroxy-2-cyclopenten-1-one
3-(1-pentyl)-2-hydroxy-2-cyclopenten-1-one

These compounds will be available for subjective evaluations following analysis for purity. The synthesis of these compounds is currently being investigated to improve yields.

(4) The ready availability of acylpyrazines has led to an investigation of their reactivity with Wittig-type reagents and Grignard reagents. In both cases, low yields of the expected product were realized, possibly because of competing nucleophilic attack on the pyrazine ring.

PROJECT NUMBER: 2525
PROJECT TITLE: Tobacco Chemistry
PROJECT LEADER: R. R. Izac
PERIOD COVERED: October, 1988

I. NATURAL PRODUCTS CHEMISTRY

- A. Objective: To isolate, identify and/or analyze natural compounds with major emphasis on tobacco and tobacco products.
- B. Results: 1.) A sample of nicotine was isolated from Premier flavor beads and purified for T. Sanders. The nicotine was identified as natural or 1- nicotine by nmr (R. Bassfield), optical rotation (J. Paine) and ORD (T. Howell). 2.) Samples of cigarette paper, which contained spots were analyzed for solanesol by TLC. Dichloromethane extracts of all samples looked identical, containing solanesol as a major component and other tobacco components. A second batch of Art tobacco and a sample of the contents of a dry ice trap collected from treated tobacco (samples obtained from S. Wren) were analyzed. GC (David Douglas) and TLC of the tobacco indicated that there wasn't any observable difference between the treated and control tobaccos. The material collected from the trap was centrifuged to yield a small amount of wax and further extracted with dichloromethane to yield a small amount of tobacco volatiles. The remaining aqueous material contained an amount of propylene glycol and a lesser amount of acetic acid. These fractions were analyzed by J. Naworal using MS. 3.) As part of a collaboration with Project 6906, a number of separation schemes were discussed. One of these involved separation of Burley S1 (BuS1) fraction using a gel column (TSK Toyopearl HW 40F). A MPLC column was prepared and about five grams of BuS1 was separated into two fractions.
- C. Plans: Continue analysis of causative agent from the Art tobacco cigarette paper and continue separation of BuS1.
- D. References:
1. Izac, R. Notebook No. 8632.
 2. Core, M. Notebook No. 8608.

II. LOW NICOTINE

- A. Objective: To examine characteristics of unextracted nicotine.
- B. Results:
- 1.) Grafted tobacco on a tomato rootstock appears to contain only extractable nicotine. Earlier grafts did not take and four more grafts were prepared.
- C. Plans: Continue to investigate genetic and cultural factors which influence the amount of unextracted nicotine in tobacco materials.

D. References:

1. Izac, R. Notebook No. 8632.
2. Bass, R. Notebook No. 8607.
3. West, G. Notebook No. 8559.

III. GREENHOUSE STUDIES

- A. Objective:** To maintain the R&D greenhouses, to conduct plant research studies and to provide greenhouse-grown tobacco materials for support of other R&D programs.
- B. Results:** 1.) The hydroponic Burley 21 plants of Group 15 are showing good growth. All preparations for Group 16 including the transplanting of Burley 21 plants have been made. These plants are being grown for Project 1904. 2.) The Burley 21/*N. glutinosa* grafts were harvested. The purpose of this is to provide a plant which boosts the production of nornicotine. 3.) The greenhouse maintenance operations including seeding, transplanting, nutrient solution preparation and other cultural tasks were completed. 4.) The experimental flue-cured and Oriental tobacco grown at Whiteville Research Station are still being cured.
- C. Plans:** Maintain production of fresh root tissue by hydroponic culture. Monitor the field plots as needed.
- D. References:**
1. Bass, R. Notebook No. 8607.
 2. West, G. Notebook No. 8559.

IV. SUPPORT ACTIVITIES

- A. Objective:** To provide requested assistance for special projects.
- B. Results:** 1.) An investigation into the synthesis of poly(isopropenyl alcohol) was started. Poly(isopropenyl acetate) was reacted with freshly prepared sodium methoxide in methanol to give a product which is consistent with poly(isopropenyl alcohol). poly(isopropenyl alcohol) was reacted with menthyl chloroformate to prepare poly(menthyl isopropenyl carbonate), polymic, by another route. 2.) The 1988 SC Flue-Cured variety evaluation display and the NC Flue-Cured variety evaluation display were attended. Tobacco leaf was evaluated in conjunction with PM Leaf Department personnel. Additional samples of "cherry red" tobacco were obtained for evaluation. 3.) Seven handmade cigarettes containing a central core of balsa wood were fabricated and will be compared to cigarettes made previously from pith wood.
- C. Plans:** Continue to investigate preparation of poly(isopropenyl alcohol) and polymic.
- D. References:**
1. Izac, R. Notebook No. 8632.
 2. Bass, R. Notebook No. 8607.

PROJECT NUMBER: 6502
PROJECT TITLE: Environmental Tobacco Smoke
PROJECT LEADER: C. E. Thomas
PERIOD COVERED: October, 1988

I. EFFECT OF GLYCERINE ON MS AND SS ACROLEIN DELIVERY

A. Objective: Determine, using the tunable diode laser system (TDL), the MS and SS acrolein deliveries on cigarettes made with varying levels of glycerine.

B. Results:

MF blend cigarettes were obtained from Flavor Development which had different concentrations of glycerine in their filler. A large data base which included other cigarette models have been analyzed but for clarity, only results from two models are given in the table below (1).

<u>MF Blend Cigt.</u> <u>(Glycerine %)</u>	<u>MS Acrolein</u> <u>(mg/cigt.)</u>	<u>SS Acrolein</u> <u>(mg/cigt.)</u>
Low Gly. A/C and Casing, (0.8%)	0.10	0.56
Std. MF Blend A/C And Casing, (2.3%)	0.12	0.64

C. Conclusions: It was found in this study that reducing the glycerine levels in a otherwise standard MF blend cigarette reduced the delivery of acrolein in the SS smoke. The MS acrolein deliveries were reduced as well, but not to the extent observed in the SS. This work is in excellent agreement with work reported by C. Blake [2].

D. Plans: The cigarettes used in this study will analysed by HPLC procedure for smoke aldehydes. Acrolein, formaldehyde, acetaldehyde, butyraldehyde, and crotonaldehyde deliveries in MS and SS will be measured and compared to the glycerine content of the blends.

E. References:

1. Parrish, M., PM Notebook #8617, pp. 99-100.
2. Blake, C., "The Effect of the Humectants Glycerol and Propylene Glycol in Mainstream and Sidestream Smoke Deliveries of Acrolein, Formaldehyde, Acetaldehyde, Acetone, and Propionaldehyde," February 1987, FTR, Neuchatel.

II. ANALYSIS OF MS SMOKE OF ART BLEND CIGARETTES

A. Objective: Measure deliveries of ammonia and acrolein in the MS smoke of ART and control cigarettes.

- B. Results: The acrolein delivery of ART cigarettes was measured by both the TDL and the HPLC method for aldehydes [1,2]. Both procedures found 100 ug/cigt of acrolein delivered in the MS smoke of the ART and 100 ug/cigt delivered in the control. The MS ammonia delivery of the ART cigarettes was determined using the ion chromatography procedure and was found to be 130 ug/cigt [3]. The ammonia delivery of the control was 35 ug/cigt. In addition to total MS ammonia, the ammonia deliveries of the eighth puffs of the ART and control cigarettes were also measured. The eighth puff of the ART cigarette delivered 12-15 ug of ammonia compared to 4 ug for the control.
- C. Conclusions: There are no differences in acrolein deliveries between the control and the test ART cigarettes. However, the ART cigarettes deliver about three times the MS ammonia of the control. In a previous study [4], it was found that the threshold (level of ammonia needed to be detected by 50% of a smoking panel), was about 20 ug/puff. The ART cigarettes are approaching this threshold. In addition, the increased alkalinity of the smoke may also be affecting the deliveries of other basic compounds.
- D. Plans: Additional analyses of the MS smoke from ART cigarettes will be conducted. These include benzene, toluene, other aldehydes besides acrolein, and the ketones. The studies on the ammonia deliveries will be repeated and will include additional samples that have been provided by B. Handy.
- E. References:
1. Lipscomb, J., PM Notebook #8703, p. 5.
 2. Thomas, E., PM Notebook #8502, p. 79.
 3. White, G., PM Notebook #8088, pp. 10-11.
 4. Parrish, M., Harward, C., and G. Vilcins, "Determination of the Gaseous Ammonia Threshold in Cigarette Smoke Part I. Analytical Methodology," Completion Report, June 7, 1985, Acc. No. 85-175.

PROJECT NUMBER: 6505
PROJECT TITLE: Special Investigations/Methods Development
PROJECT LEADER: D. F. Ingraham
PERIOD COVERED: October, 1988

I. PROJECT ART

- A. Objective: Provide analytical support to project ART.
- B. Results: A problem was noticed last month in which results of nicotine determinations from ART extracted tobacco were different on different models of gas chromatographs. After several weeks of investigations, similar results are now being obtained while research continues on what may have led to these observed differences. In addition, an independent HPLC method is currently under development to provide a referee method for the determination of nicotine from extracted tobaccos.
- C. Plans: Continue analytical support on an as needed basis. Finish development of the HPLC method and continue research into the discrepancies observed between different GC's.

II. ANALYSIS OF RESIDUAL SOLVENTS IN PACKAGING MATERIAL

- A. Objective: To provide headspace analyses for residual solvents from packaging materials and develop a QA method for the routine analysis of packaging materials.
- B. Results: QA continues to analyze routine packaging material for residual solvents while Project 6505 is analyzing samples from new products and other non-routine requests such as inks. Good agreement was obtained between QA and Special Investigations. Training is being provided to some of the suppliers of packaging materials in the setup and execution of the headspace/GC method.
- C. Plans: We will continue to analyze non-routine samples and provide support to QA as needed.

III. MATERIALS EVALUATION

- A. Objective: To identify components of commercial products prior to their use at PM facilities.
- B. Results: Samples analyzed this month included vacuum hoses, rollers, pipe markers, bug traps, and seals.
- C. Plans: Materials evaluation is a continuing program.

IV. RESPONSE TO ANALYTICAL REQUESTS

- A. Objective: To provide analytical support to R&D and Operations personnel and projects.

B. Results:

Analyses and investigations by project personnel during the month of October included:

Five customer complaint samples were analyzed for possible contaminants.

The amount of nornicotine in a tobacco extract was confirmed by GC/MS.

PROJECT NUMBER: 6906.
PROJECT TITLE: Biological Effects of Smoke
PROJECT LEADER: J. M. Penn
WRITTEN BY: B. D. Davies and G. M. Nixon
PERIOD COVERED: October, 1988

I. INHIBITION OF EGF BINDING ASSAY

- A. Objective: Compare ^{125}I -EGF binding in new (early passage) versus old (late passage) BALB 3T3 cell cultures in an effort to establish the new cells for assay use.
- B. Results: EGF binding kinetics were evaluated in parallel using old and new cells. Since the kinetic parameters and the response to catechol and 2R1 CSC were comparable, the new culture will become the current cell culture utilized in the assay. A memo describing this standard procedure for evaluating new cultures is in progress.
- C. Plans: Examine and compare the binding kinetics of 3T3 cells in the presence of catechol and 2R1 CSC.
- D. Reference: Patskan, G. Notebook No. 8710, p. 143.

II. GLUTATHIONE DEPLETION ASSAY (GDA)

- A. Objective: To determine if CDNB can be used as a control compound to examine the effect of 2R1 CSC induced reduction in GSH levels on TA98 mutagenic activity.
- B. Results: Two experiments were conducted to determine the effects of CDNB pretreatment on GSH levels and S/M activity in TA98. At a dose of 100 μM , CDNB produced a 100% reduction in the levels of GSH. However, when tested using a dose range including 0.1, 1.0, 10 μM , CDNB had no effect on GSH levels. Pretreatment with CDNB had no effect on S/M activity in either experiment.
- C. Plans: Experiments will be conducted to examine the pretreatment effects of a dose range of CDNB of 10 to 100 μM on GSH levels in TA98. In addition, an experiment will be conducted to determine the effects on S/M activity of the same dose range plated with TA98 cells.
- D. Reference: McCoy, W. R. Notebook No. 8555, pp. 183-187.

III. NICOTINE SPECIFIC MONOCLONAL ANTIBODY

- A. Objective: To obtain a monoclonal antibody (MCA) against nicotine (NIC-MCA).
- B. Results: Following an immunization boost, another series of sera was collected and tested by the contract lab for reaction to the

antigen. Results from that analysis indicate an increase in titer for all mice. These samples are also being sent to PM for further analysis.

- C. **Plans:** Upon receipt of the preparation of enzyme labeled anti-mouse antibody, these samples will be tested here at PM. In addition, we are attempting to obtain additional quantities of the reactive succinylated hydroxyethylnicotine to prepare additionally required antigens.
- D. **Reference:** Davies, B. D. Notebook No. 8638, p. 90.

IV. BINDING OF ^3H -PDBU TO 3T3 CELLS

- A. **Objective:** Examine the effects of CSC and catechol on the binding of ^3H -PDBU to 3T3 cells.
- B. **Results:** Three preincubation (19.5 hr.) experiments were performed using catechol at levels ranging from 0.5 $\mu\text{g}/\text{ml}$ to 5 $\mu\text{g}/\text{ml}$ with and without 100 $\mu\text{g}/\text{ml}$ 2R1 CSC. Catechol alone and CSC alone both increased binding in a dose-dependent manner. At doses of 0.5 and 1 $\mu\text{g}/\text{ml}$, catechol had an additive effect with CSC. At higher doses, the combined effect was less than additive, and the response to catechol appeared to plateau.
- C. **Plans:** Saturation curve experiments will be performed using 200 $\mu\text{g}/\text{ml}$ 2R1 CSC for preincubation and varying doses of ^3H -PDBU at time of binding.
- D. **Reference:** Burruss, T. J. Notebook No. 8730, p. 20.

V. PROTEIN KINASE C (PKC) IN INTACT CELLS

- A. **Objective:** To determine the dose response of 3T3 cells to fresh 2R1 CSC in the PKC intact cell assay.
- B. **Results:** Four dose response experiments were conducted using fresh 2R1 CSC at levels ranging from 50 $\mu\text{g}/\text{ml}$ to 250 $\mu\text{g}/\text{ml}$. Experiments conducted according to standard protocol showed inconsistent results on autoradiography. There was variability among triplicate samples as to which dose induced maximal phosphorylation increases. Two dose response experiments were conducted with a trichloroacetic acid (TCA) precipitation step incorporated prior to solubilization, and both experiments showed a qualitative dose response to fresh 2R1 CSC. The phosphorylation response to fresh 2R1 CSC was maximal at a dose of 200 $\mu\text{g}/\text{ml}$.
- C. **Plans:** Test the dose response of 3T3 cells to frozen 2R1 CSC.
- D. **Reference:** Nixon, G. M. Notebook No. 8711, p. 29.

VI. SALMONELLA/MICROSOME (S/M) ASSAY

- A. Objective: To evaluate the effects of the addition of d-alanine to 1R4F filler using water as the solvent.
- B. Results: An experiment was conducted in which 0.5, 1.0 and 2.0% d-alanine dissolved in water was oversprayed onto 1R4F filler. The S.A.s of the resulting IT CSCs were determined. The S.A.s of the 1R4F untreated control and the solvent control (water) were not statistically different from each other. All concentrations of d-alanine added to 1R4F filler increased the IT CSC S.A.s as compared to that of 1R4F alone. However, only the 2% level of added d-alanine produced IT CSC S.A.s that were significantly higher than the solvent control.
- C. Plans: To test d-alanine at 446 and 892 ppm on 1R4F filler.
- D. Reference: Thompson, L.H. Notebook No. 8628, p. 197.

PROJECT NUMBER: 6908
PROJECT TITLE: Smoke Condensate Studies
PROJECT LEADER: A. H. Warfield
PERIOD COVERED: October, 1988

I. TSNA PRECURSORS

- A. Objective: To determine the precursors of MS TSNA.
- B. Results: Work continues on preparation of a suitable form of a nicotine salt for use as a model of bound nicotine. Attempts to use a SPEX Freezer/Mill to grind nicotine bitartrate crystals to the particle size desirable for subsequent microencapsulation showed that this procedure produces too low a throughput to be practical. Two large mortars have been procured to enable adequate material to be ground using a more conventional approach.

A nicotine amino acid ester has been prepared as a possible nicotine release compound to act as a model for bound nicotine. The compound, *t*-butyl 2-(3-pyridinyl)-1-pyrrolidinylacetate, is an analogue of an ethyl ester prepared by G. Chan earlier.

Sugars have been added to burley (Bu) CEL to simulate the level of sugars present in oriental (Or) CEL, and an RL prepared by spraying this mixture on Bu base web (BW). Cigarettes made from this RL will be smoked for TSNA determination. This experiment was designed as a result of data reported previously where Or + Bu CEL's gave reduced TSNA relative to burley CEL alone when both were added to BuBW and corresponding cigarettes smoked. FTIR-EGA studies have also showed that ammonia evolutions from BuCEL/BuBW were eliminated when OrCEL was mixed with BuCEL before the RL was prepared. It is postulated that sugars in OrCEL react with soluble ammonia in BuCEL to form amino sugars, which act as scavengers for nitrosating agents that cause pyrosynthesis of MS smoke TSNA.

An experiment was carried out to determine conditions necessary to extract nicotine from uncured tobacco leaves in order to allow the tobacco to be cured after removal of nicotine. This activity is being carried out to determine whether removal of nicotine before curing will result in lowering of MS smoke NNK from the cured tobacco. Greenhouse burley leaves obtained from R. Bass were divided in half along the midvein, and 5-6 half leaves were extracted with 2 L hexane or 5% ethanol in hexane, while some were retained as a control. The extractions were carried out overnight. Each sample was extracted with water in a blender, and the alkaloids partitioned into methylene chloride at pH 10. Nicotine analysis (GC/MSD) showed that hexane removed 35% of the nicotine while 5% ethanol/hexane removed 52%. Ripe burley leaves were obtained from J. Smiley, University of Kentucky (via David Connors). Ten leaves were extracted with 3 L of 5% ethanol/hexane on a shaker for 3 days, followed by evaporation of residual solvent and air-curing of the leaves. Ten additional leaves were treated in the same manner, and the solvent changed, followed by

an additional overnight extraction before curing. The control (10 leaves) was also subjected to air-curing.

- C. Plans: When correctly sized nicotine bitartrate particles are obtained, submit samples for microencapsulation, and add to appropriate fillers. Add the nicotine amino acid ester to filler. Determine the effects of these additions on MS TSNA deliveries. Smoke the BuCEL + sugar/BuBW cigarettes and determine TSNA for comparison to Bu + Or CEL/BuBW results. After the burley samples are air-cured for 7 weeks or more, they will be stemmed, cut, cigarettes prepared, and both filler and MS smoke analyzed for TSNA. The filler will also be analyzed for nicotine.

D. References:

Haut, S. A. Notebook 8595, p. 164.
Morgan, W. R. Notebook 8579, p. 70.
Warfield, A. H. Notebook 8558, pp. 193-195.

II. ANALYTICAL PROCEDURES

- A. Objective: To develop and maintain analytical methodology for TSNA or other compound classes where information is needed for determining relationships of TSNA to their precursors.
- B. Results: Method development for simultaneous VNA/TSNA determination on the same smoke sample was continued. Addition of the VNA fraction to the TSNA fraction caused rapid deterioration of the chromatographic system. Addition of a precut to the alumina cleanup step failed to remove the cause of this problem. Further modifications to the method are being investigated.

Efforts were made to establish a GC/MSD method for nicotine and minor alkaloids. However, the approach utilized did not result in consistently reproducible response factors for these compounds. Therefore, a previously reported GC/NPD method using KOH/methanol extracts is now being reestablished. In this method, the internal standard and all components measured have the same response factor.

- C. Plans: Continue development of the VNA/TSNA method. Finish the reestablishment of the GC/NPD method for alkaloids and use it to analyze Project ART samples.

D. References:

Morgan, W. R. Notebook 8579, p. 70.
Tickle, M. H. Notebook 8716, pp. 28-29.
Levins, R. J. Notebook 8672, p. 80.

III. SUPPORT FUNCTION: CONDENSATE PREPARATION

- A. Objective: To fabricate cigarettes, perform smokings, and prepare condensate as needed for biological and chemical analysis.
- B. Results: Eleven samples prepared as part of the Crossed Solubles Base Web Study were sprayed on the specified fillers, and cigarettes made and smoked for S/M testing, along with the appropriate controls. Another RL sample based on BuBW (see I. above) was prepared from BuCEL + sucrose, fructose and glucose. Two other special cigarette samples were prepared and smoked for S/M testing.
- C. References:
- Hellams, R. D. Notebook 8613, p. 115.

PROJECT NUMBER: 8101
PROJECT TITLE: Cigarette Testing Services Division
SECTION LEADER: Joyce F. Stargardt
PERIOD COVERED: October, 1988

I. MARKET ACTIVITY

A. Objective: To monitor and report new brand introductions and brand modifications for the domestic and international cigarette markets.

B. Results:

1. Domestic

a. Premier

Premier 80 (Box) and Premier Menthol 80 (Box) from R. J. Reynolds were analyzed in our laboratories. The tobacco in these cigarettes is heated rather than burned, therefore the tar is quite different from that of a conventional cigarette. "Tar" is referred to as nicotine-free dry particulate matter (NFDPM) and is obtained by subtracting nicotine and water from the TPM. Cigarettes were lit by preheating statically on-line for 30 seconds with a Scripto Lighter until the first puff was taken. Some of the analytical data for Premier is listed below. More information can be found in a memo to J. E. Wickham entitled "Premier Cigarettes (R.J. Reynolds)".

	Premier 80 (Box)	Premier Menthol 80 (Box)
<u>Smoke</u>		
NFDPM, mg/cigt.	3.0	2.5
TPM, mg/cigt.	6.7	5.4
Nicotine, mg/cigt.	0.20	0.15
Water, mg/cigt.	3.53	2.77
Puffs/Cigt.	10.3	10.1
Menthol, mg/cigt.	ND	0.10
CO, mg/cigt.	10.0	10.4
Glycerine, mg/cigt.	2.9	2.9

b. New Brands

Brown & Williamson is test marketing Capri 120 Box (plain and menthol) cigarettes in New Orleans, LA. These cigarettes deliver 12 mg tar and 0.9 mg nicotine. The smoke menthol delivery is 1 mg. These cigarettes like Capri 100, have a 17 mm circumference.

The Liggett Group has introduced Eve Ultra 120 Box (plain and menthol) cigarettes. These cigarettes deliver 5 mg tar and 0.5 mg nicotine. The menthol smoke delivery is 0.4 mg. This is a brand extension of Eve Lights 120 currently on the market. Both cigarettes have a 21 mm circumference.

R. J. Reynolds has introduced new package graphics on Winston 80, 85 and 100, Winston Lights 85 and 100 and Winston Ultra 85 and 100 cigarettes. The eagle symbol is more prominent on the new package. During the past year Reynolds has changed the graphics on the Winston, Vantage, Salem, Doral and Century products. No changes were noted in the smoke deliveries of these cigarettes.

2. International

Japan Tobacco, Inc. introduced Caster Mild King Size cigarettes to the Japanese market on October 1, 1988. This brand delivers 6 mg tar and 0.6 mg nicotine. This makes three versions of Caster on the market in Japan.

R. J. Reynolds introduced Island Super Lights 85 in Japan on October 1, 1988. This brand delivers 8 mg tar and 0.6 mg nicotine. These product, like Island Lights 85, contains freon expanded tobacco.

Kent Special Milds King Size cigarettes were introduced in Japan on October 1. This brand is manufactured by Brown & Williamson for export to Japan. This brand delivers 10 mg tar and 0.8 mg nicotine and is the third version of Kent on the market in Japan.

II. ANALYTICAL METHODS DEVELOPMENT AND SUPPORT

A. Objective: To evaluate and recommend analytical methods and new technology in support of programs for R&D and Manufacturing.

B. Results:

The relationship of firmness to oven volatiles was analyzed at five relative humidity levels (40%, 50%, 60%, 70% and 75%) and 75°F for Marlboro 85 cigarettes. The resulting firmness "correction factor" was 0.39 mm, firmness/% OV for cigarettes manufactured at the Manufacturing Center in Richmond, Virginia. This number compares favorably with the 0.36 mm/% value reported by W. L. Mokarry in 1983. ("Cigarette Firmness Correction Factor," Inter-Office Correspondence, October 14, 1988, To J. E. Wickham)